

AUSTRALIA
Patents Act 1990

NOTICE OF ENTITLEMENT

I/We,PERNOD RICARD.....

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being the applicant in respect of Application No. 11118/95, state the following:-

Must be completed for all applications:

The person(s) nominated for the grant of the patent:

~~is/are the actual inventor(s)~~

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has, for the following reasons, gained entitlement from the actual inventor(s):

by a contract of employment between the applicant and the inventor..

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Must be completed if the application was made under the PCT

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is/are the applicant(s) of the application(s) listed in the declaration under Article S of the PCT

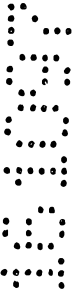
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The basic application(s) listed in the declaration made under Article S of the PCT

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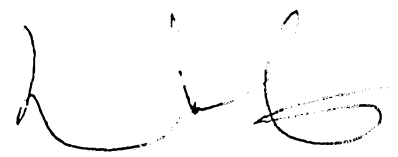
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Deposit List (by number, deposit institution, date)

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Dated 14/5/97



Mr. CHATEAUNEUF - Director

Please type name and position of signatory





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(12) PATENT ABRIDGMENT (11) Document No. AU-B-11118/95
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PROCESS FOR WASHING REFILLABLE PLASTIC BOTTLES
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- (56) Prior Art Documents
EP 479393
US 4680060
WO 91/17236
- (57) Claim

1. A process for washing a container, especially a food or drink container, of the bottle type and intended to be reused, especially for a use with foodstuffs, in which:

- the reusable container is made of a homopolymer or a copolymer having an increased resistance to the binding of organic compounds or a mixture of said polymers, which is transparent and which has an average degree of crystallinity higher than 30%, the thickness of the walls of the container being greater than:
 - 0.35 mm with regard to the body
 - 1.00 mm at the base
 - 1.00 mm at the bottom, and
- said container is washed with an aqueous solution at a temperature higher than 70°C.

OPI DATE 13/06/95 APPLN. ID 11118/95
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<p>(51) Classification internationale des brevets ⁶ : C11D 11/00</p>	<p>A1</p>	<p>(11) Numéro de publication internationale: WO 95/14761 (43) Date de publication internationale: 1er juin 1995 (01.06.95)</p>
<p>(21) Numéro de la demande internationale: PCT/FR94/01361 (22) Date de dépôt international: 22 novembre 1994 (22.11.94) (30) Données relatives à la priorité: 93/13927 22 novembre 1993 (22.11.93) FR (71) Déposant: PERNOD RICARD [FR/FR]; 142, boulevard Haussmann, F-75008 Paris (FR). (72) Inventeur: ROBICHON, Patrice; 4, rue Au-Mètre, F-78290 Croissy-sur-Seine (FR). (74) Mandataire: WARCOIN, Jacques; Cabinet Regimbeau, 26, avenue Kléber, F-75116 Paris (FR).</p>	<p>(81) Etats désignés: AU, CA, CN, JP, KR, NZ, RU, brevet européen (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Publiée <i>Avec rapport de recherche internationale. Avant l'expiration du délai prévu pour la modification des revendications, sera republiée si de telles modifications sont reçues.</i></p> <p style="text-align: center;">69 1536</p>	
<p>(54) Title: PROCESS FOR WASHING REFILLABLE PLASTIC BOTTLES (54) Titre: PROCEDE DE LAVAGE DE BOUTELLES EN PLASTIQUES RE-REMPLISSABLES (57) Abstract Process for washing a container, for instance a bottle-type food container reusable for example for drinking products, characterised in that a container made of a homopolymer or copolymer or a mixture thereof is used, said container being transparent, with an average crystallinity rate of more than 30 % and a wall thickness of more than 0.35 mm for the body, 1.00 mm for the base, and 1.00 mm for the bottom; and in that the container is washed with an aqueous solution at a temperature of more than 70 °C. (57) Abrégé La présente invention concerne un procédé de lavage d'un contenant, notamment à usage alimentaire de type bouteille et destiné à être réutilisé, notamment pour un usage alimentaire, caractérisé en ce que: on utilise un contenant réalisé en un homopolymère ou un copolymère ou mélange de dits polymères, transparent, ayant un taux de cristallinité moyen supérieur à 30 %, l'épaisseur des parois du contenant étant supérieure à: 0,35 mm pour le corps; 1,00 mm à la base; 1,00 mm au fond; et on lave ledit contenant avec une solution aqueuse à une température supérieure à 70 °C.</p>		

The present invention relates to the field of refillable plastic bottles, especially for use with foodstuffs.

5 The plastic bottles employed especially for packaging carbonated beverages consist predominantly of polyethylene terephthalate (PET hereinafter).

10 Compared with other plastics (in particular PVC), PET exhibits excellent gloss and transparency properties and, compared with glass, it has the advantage of lightness and mechanical strength and this is why it is widely employed for large containers.

15 It has good mechanical properties and it offers good chemical resistance to oils, fats and the majority of organic solvents and solutions, to inorganic salt solutions and to hydrocarbons.

Thanks to biaxial stretching, the impact strength of PET is multiplied by 10 and its elasticity modulus by 4.

20 In addition, its impermeability to gases is remarkable, especially to carbon dioxide and to oxygen, which results in its very extensive use for carbonated beverages.

As a result of these properties, PET has become the reference packaging for large single-use containers.

25 However, there are many countries where refillable large containers occupy a predominant place, be it for legislative and ecological reasons (Northern Europe) or for economic reasons (Scuth America). This is why new bottles appeared in Germany in 1990, which are refillable
30 PET bottles. These bottles are a replacement for the returnable glass for the same reasons which made PET a success for single use, namely their strength and lightness.

35 The refillable bottles employed at present are made of PET by the conventional two-stage process.

This two-stage process separates the injection molding of a preform made of amorphous PET and the biaxial stretching of the preform to obtain the final container.



In the first stage the preforms are injection-molded on a press fitted with a drive and a multi-cavity mold. The preforms may be stored and transported to the point where the final bottle is blown.

5 In the second stage the amorphous PET preform is taken up, reheated to the optimum temperature and then biaxially stretched, mechanically by a metal plunger in the lengthwise direction and by high-pressure blowing in a transverse direction.

10 This process gives rise to two ranges of industrial machines:

- injection presses for preforms up to 96 cavities and
- biaxial stretch blow molders up to 40,000 bottles/hour.

15 This process represents approximately 80 % of the PET bottles manufactured worldwide, refillable or otherwise.

20 However, even after biaxial stretching, the PET bottles do not exhibit mechanical or even chemical properties (as will be seen later) which enable them to be re-employed.

25 To obtain a sufficient mechanical strength, the wall thickness must be increased and, in order to obtain refillable plastic bottles, this means doubling their weight.

The single-use bottles generally weigh of the order of 48 g in the case of a 1.5 l bottle, against approximately 100 g in the case of refillable bottles.

30 Furthermore, since these refillable bottles are manufactured by conventional technology, they must be washed at a temperature lower than 60°C; above this temperature they shrink too much and may distort.

35 However, these conditions of washing at a temperature lower than 60°C do not always make it possible to remove certain organic compounds which are bound by physical or chemical adsorption on the inner walls of the bottles, especially when the latter have been employed for storing non-foodstuff products.

This is why, on washing lines for these PET



bottles, it is necessary to provide sniffers which sample each bottle to analyse for the possible presence of a certain number of contaminating agents, in order to reject the contaminated bottles.

5 The need for these very precise controls frequently results in the rejection of a large number of refillable bottles and thereby decreases the number of cycles of each bottle, this being in addition to the bottles rejected for mechanical defects, especially
10 cracks, which, all in all, obviously appreciably reduces the profitability of the reuse process.

 Thus, it was originally envisaged that refillable PET bottles were to withstand from 15 to 25 cycles, 15 cycles being the minimum objective, whereas in reality it
15 is found that the average number of cycles undergone by a bottle is much lower than 15; 6-7 cycles are referred to in the case of some bottles, which is obviously very inadequate.

 Various organizations have been concerned with
20 the problem of refillable plastic bottles, this being in order to establish good codes of practice for companies employing refillable plastic bottles and in order to have better knowledge of the properties of these bottles when they are contaminated by foreign substances.

25 This is the case especially with the TNO (The Netherlands Organization for Nutrition and Food Research) and also with research which is conducted within the scope of the International Life Science Institute on this same type of product.

30 For the time being, the proposed solutions consist essentially in modifying the washing procedures so as to improve the decontamination, in employing more efficient sniffers and optionally in investigating new polymers or polymer blends which could make it possible
35 to obtain refillable bottles that can be washed under better conditions.

 The document EP-A-479 393 describes transparent containers of bottle type made of polyethylene

AMENDED SHEET



terephthalate having a degree of crystallinity of between 24 and 30%. These containers can be washed by a caustic washing solution up to a temperature of 60°C.

5 The present invention is based on the finding of a property of certain aromatic polyesters which is that of resisting the binding of organic compounds which are possible contaminants of plastic bottles, the said property being additionally accompanied, in the case of these bottles, by a satisfactory resistance to drastic
10 washing conditions.

The present invention is based on the use of a process making it possible to increase the resistance of aromatic polyesters, especially PET, to the binding of organic compounds, in which said polyester is treated to
15 raise its crystallinity above 30 % and preferably above 40 %.

Processes already exist which make it possible to increase crystallinity by virtue of a relaxation of stresses by annealing, especially of PET above 30 % and
20 even above 40 %; among these processes there may be mentioned especially the so-called SRF or SRCF process developed by the Sidel company and forming the subject especially of patent numbers EP 0,237,459, FR 2,649,035 and FR 2,658,119.

25 The annealing may be performed by different techniques, especially by the hot mold technique or by softening the biaxially stretched product.

The SRCF process is a particular method of implementation of the general process, in which to
30 increase the crystallinity of aromatic polyesters the polyester in polymerized form is first of all biaxially drawn and then softened so as to release the stresses induced by the biaxial stretching and then, and only then, given the definitive shape.

35 The SRCF process, which is derived from the two-stage process, starts with a preform manufactured on an independent press. The continuation of the process comprises 4 stages:



- 1) the body of the preform is heated,
- 2) a first oversized bottle is biaxially stretch-blow molded,
- 3) this bottle is passed through an infrared oven called "recovery oven"; two things happen: on the one hand the neck is crystallized and becomes white; on the other hand, under the effect of the temperature, 200°C for approximately 1 minute 30, the body of the bottle contracts: the stresses induced during the biaxial stretching stages have been released, the bottle then takes on a "potato-like" shape; the density of the PET of which the body of the article is composed has greatly increased, the PET is thereafter crystallized and has become heat-resistant,
- 4) the article is then finished by a second blowing with biaxial orientation, the bottle then taking on its definitive shape.

The previous SRF process did not include heating in an infrared oven; the ring was crystallized before the reheating of the preform.

This process has already been employed so as to improve the heat resistance of single-use bottles, this being in order to permit hot filling with a still product of pasteurization of a carbonated product after filling the bottle.

No mention has ever been made of the fact that this process increased the resistance of the polymer to the binding of organic products by physical or chemical adsorption, especially of contaminating products that can be found in refillable bottles which have been employed for storing household products, chemicals or other products.

This is why the present invention also relates to a process for washing a container, especially a food or drink container, of the bottle type and intended to be reused, especially for a use with foodstuffs, in which:

- the reusable container is made of a homopolymer or a copolymer having an increased resistance to the binding of organic compounds or a mixture of polymers, which is



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4 March 1998

transparent and which has an average degree of crystallinity higher than 30 %, the thickness of the walls of the container being greater than:

- 0.35 mm with regard to the body
- 5 - 1.00 mm at the base
- 1.00 mm at the bottom, and
- said container is washed with an aqueous solution at a temperature higher than 70°C.

The plastic containers employed in this process have the characteristic of being capable of being washed under conditions close to the conditions employed for returnable glass bottles, and the said containers, being thicker than the non-reusable bottles, exhibit mechanical characteristics which ensure their refilling under good conditions.

In particular, this type of container can be washed with an aqueous solution containing a base, especially a hydroxide at concentrations which can vary from 1 to 3 % in the presence of a wetting agent, this washing being optionally carried out at temperatures higher than 80°C and possibly reaching 85°C, temperatures and conditions that are usually employed for reusable glass containers.

The container is preferably made of a polymer or a copolymer of polyester type, or a mixture of such polymers, involving in particular linear polyesters resulting from the condensation of an aromatic diacid and a diol.

Among the polymers which can be employed there should be mentioned the polymers obtained with:

- 30 - terephthalic acid
- naphthalic acid
- dimethyl terephthalate
- isophthalic acid
- dimethyl isophthalate
- 35 by condensation with diols such as:
 - ethylene glycol
 - 1,4-cyclohexanedimethanol.

Among the different possible polymers or copolymers, there must be mentioned, very particularly,



PET and the polyethylene glycol naphthalate copolymer (PEN hereinafter), as well as mixtures of these two copolymers (PET + PEN blends).

5 In this process, it will be preferred to employ bottles with relatively thick walls while keeping the weight advantages linked with the use of plastic bottles; in particular, the container walls will be able to have thicknesses greater than:

- 10 - 0.4 mm with regard to the body (or even greater than 0.5 mm)
- 1.25 mm at the base (or even greater than 1.7 mm)
- 1.90 mm at the bottom (or even greater than 2.5 mm)

15 In order to ensure an equal distribution of the stresses induced by the hot washing, it will be preferred to employ containers which have substantially equal wall thicknesses at both the body and the base, and to employ, for example, a container which has a substantially hemispherical bottom, by then making use of a known technique of adhesively bonded basecup to enable the
20 bottle to stay upright.

Nevertheless, it is possible to envisage self-stable or petaloid bottoms.

The present invention preferably relates to containers comprising a label that can be unstuck under
25 the washing conditions, this being so as to enable the container to be washed inside and outside all at the same time in the washing solution.

The container according to the present invention is preferably obtained by making use of one of the
30 processes described above.

The present invention also relates to the containers intended to be washed by the process described above.

35 Other characteristics and advantages of the present invention will appear on reading the example below.

EXAMPLE

Two types of bottles were tested, exhibiting crystallinity percentages lower than 10 % and higher than



40 %.

Bottles of the first type are obtained by conventional techniques in two stages with control of the crystallinity. Bottles which have a degree of crystallinity higher than 40 % are obtained by the SRCF process referred to above.

The contaminants employed are:

- methyl parathion diluted to 0.4 g/l,
(this compound being that presenting the main problem of decontamination according to the TNO analyses) and
- lead-free 98 premium gasoline, undiluted,
(this compound being the contaminant most frequently stored in refillable bottles).

The contaminant is stored in the bottle for 48 hours before the washing.

The washing solution employed is a solution of sodium hydroxide (NaOH) at a concentration of 1.5 % with a liquid P3 Stabilon detergent at a concentration of 0.3 %.

The following are performed:

- a pre-rinsing by injecting hot water ($45 \pm 5^\circ\text{C}$) for 2 minutes,
- then a washing at 60 or 80°C with the washing solution for 10 minutes,
- finally, rinsing by injecting hot water ($45 \pm 5^\circ\text{C}$) for 2 minutes and injecting cold water for 2 minutes.

The results observed are as follows:



TABLE 1

Degree of residual contamination by parathion-methyl in $\mu\text{g}/\text{bott}$ as a function of the washing temperature

	After washing at 60°C	After washing at 80°C
Percentage of crystallization of the bottle	Cumulative total in the material and the simulant product	Cumulative total in the material and the simulant product
$\geq 40 \%$	1.4	ND
$\leq 10 \%$	2.5	-

ND = No. Detected

TABLE 2

Degree of residual contamination by gasoline in mg/bott as a function of the washing temperature

	After washing at 60°C	After washing at 80°C
Percentage of crystallization of the bottle	Cumulative total in the material and the simulant product	Cumulative total in the material and the simulant product
$\geq 40 \%$	4.60	0.39
$\leq 10 \%$	6.42	-

It is found in Table 1 that at 60°C the bottles according to the invention exhibit nearly 50 % less of contaminants when compared with the traditional reusable bottles.

In Table 2 a decrease of 30 % in the contamination is similarly found in the case of the bottles according to the invention when compared with traditional reusable bottles washed at 60°C.

However, of course, the temperature-resistant



bottles according to the invention can be washed at 80°C, which is not the case with traditional bottles. In this case, the residual contamination (after washing at 80°C) is reduced by 91 % when compared with washing at 60°C and
5 by 94 % when compared with traditional bottles (Table 2).

In Table 1 the residual contamination of the bottles according to the invention (washed at 80°C) is below the threshold of detection.



The claims defining the invention are as follows:

1. A process for washing a container, especially a food or drink container, of the bottle type and intended to be reused, especially for a use with foodstuffs, in which:

- 5 - the reusable container is made of a homopolymer or a copolymer having an increased resistance to the binding of organic compounds or a mixture of said polymers, which is transparent and which has an average degree of crystallinity higher than 30%, the thickness of the walls
10 of the container being greater than:
- 0.35 mm with regard to the body
 - 1.00 mm at the base
 - 1.00 mm at the bottom, and
- said container is washed with an aqueous solution at
15 temperature higher than 70°C.

2. The process as claimed in claim 1, wherein the aqueous solution contains a base and a wetting agent.

3. The process as claimed in claim 1 or claim 2, wherein the aqueous solution is a solution with at least 1% by weight of
20 hydroxide.

4. The process as claimed in one of claims 1 to 3, wherein the temperature is higher than 80°C.

5. The process as claimed in one of claims 1 to 4, wherein the polymer is a polyester.

25 6. The process as claimed in one of claims 1 to 5, wherein the container is made of a homopolymer or a copolymer or a mixture of said linear polymers, of an aromatic diacid and of a diol.



7. The process as claimed in claim 6, wherein the container is made of a homopolymer or a copolymer or a mixture of said polymers of terephthalic acid or a naphthalic acid and of ethylene glycol.

8. The process as claimed in claim 7, wherein the container is a homopolymer or a polyethylene glycol terephthalate or polyethylene glycol naphthalate copolymer or mixtures thereof.

9. The process as claimed in claim 8, wherein the container is a mixture of polyethylene glycol terephthalate with from 5 to 15% by weight of polyethylene glycol naphthalate.

10. The process as claimed in one of claims 1 to 9, wherein the average degree of crystallinity is higher than 40%.

11. The process as claimed in claim 10, wherein the glass transition temperature is greater than or equal to 90°C.

12. The process as claimed in one of claims 1 to 11, wherein the container is subsequently rinsed with softened water whose temperature is less than approximately 30°C.

13. The process as claimed in one of claims 8 to 11, wherein the container has a thickness greater than:

- 0.4 mm with regard to the body
- 1.25 mm at the base
- 1.90 mm at the bottom.

14. The process as claimed in one of claims 1 to 13, wherein the container has a substantially hemispherical, self-sealing or petaloid bottom.



15. The process as claimed in one of claims 1 to 14, wherein the container comprises a label which can be unstuck under the washing conditions.

16. The process as claimed in one of claims 1 to 15, wherein the container is obtained from a preform by at least one biaxial stretching stage and then, after an
5 annealing to release the stresses induced, given the final shape with a degree of crystallinity higher than 30%.

17. The process as claimed in claim 16, wherein the annealing is obtained by the hot mold technique.

18. The process as claimed in claim 16, wherein the annealing is obtained by
10 softening the biaxially drawn product.

19. The process as claimed in one of claims 1 to 18, wherein the thickness of the container is substantially uniform over the body and the base.

20. A container when used in the process as claimed in one of claims 1 to 19,
15 which is made of a copolymer or mixture of copolymers of polyester type of transparent type which has an average degree of crystallinity higher than 30%, the thickness of the walls of the container being greater than:

- 0.35 mm with regard to the body
- 1.00 mm at the base
- 1.00 mm at the bottom

20 21. The container as claimed in claim 20, which is made of aromatic polyester having an increased resistance to the binding of organic compounds and obtained by treating the polyester to increase its crystallinity above 30%.



22. The container as claimed in claim 21, wherein said polyester is treated to increase its crystallinity above 40%.

23. The container as claimed in claim 21 or claim 22, wherein the polyester in polymerized form is first of all biaxially stretched, blow molded and then, after annealing to release the stresses induced, is given the final shape.

24. The container as claimed in claim 23, wherein the polyester is annealed by the hot mold technique.

25. The container as claimed in claim 23, wherein the polyester is annealed by softening the biaxially drawn product.

26. Processes for washing containers or containers when used in said processes, substantially as hereinbefore described with reference to the example.

DATED: 4 March 1998

CARTER SMITH & BEADLE

Patent Attorneys for the Applicant:

PERNOD RICARD



DA: NP #20742 RS2

4 March, 1998

INTERNATIONAL SEARCH REPORT

International Application No
PCT/FR 94/01361

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D11/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO, A, 94 01268 (CONTINENTAL PET) 20 January 1994 see page 7, line 5 - line 24 see page 15, line 18 - line 21; claims 16, 20, 23; figures	1, 6-8, 14
X	& DATABASE WPI Derwent Publications Ltd., London, GB; AN 93-367277 & TW, A, 210 976 (CONTINENTAL PET) 11 August 1993 see abstract	1, 6-8, 14, 20-25
A	EP, A, 0 479 393 (CONTINENTAL PET) 8 April 1992 see the whole document	1-8, 13-25
	-/--	

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
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- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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- *&* document member of the same patent family

Date of the actual completion of the international search

20 March 1995

Date of mailing of the international search report

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Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

Intern. Pat. Application No

PCT/FR 94/01361

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,4 680 060 (A.S. GUPTA ET AL) 14 July 1987 see claims 10-15; example 1 ---	1,2,5-8, 14
A	WO,A,91 17236 (HENKEL) 14 November 1991 see page 6, line 18 - line 23; claims ---	1,5-8,14
A	DATABASE WPI Section Ch, Week 8402, Derwent Publications Ltd., London, GB; Class A35, AN 84-011756 & ZA,A,8 207 598 (HALL A R M) 21 July 1983 see abstract ---	1,5-8
A	DATABASE WPI Section Ch, Week 9326, Derwent Publications Ltd., London, GB; Class A23, AN 93-208695 & JP,A,5 132 041 (TOYO SEIKAN KAISHA LTD) 28 May 1993 see abstract ---	1,4-8
P,X	DATABASE WPI Section Ch, Week 9403, Derwent Publications Ltd., London, GB; Class A23, AN 94-022552 & JP,A,5 330 535 (NISSEI ASB KIKAI KK) 14 December 1993 see abstract -----	1,6-8, 14,20

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/FR 94/01361

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A-9401268	20-01-94	AU-B- 4664293	31-01-94
		CN-A- 1092352	21-09-94
EP-A-0479393	08-04-92	US-A- 4755404	05-07-88
		US-A- 4725464	16-02-88
		AU-B- 606341	07-02-91
		AU-A- 7368087	03-12-87
		CA-A- 1304301	30-06-92
		DE-A- 3774885	09-01-92
		DE-A- 3784965	29-04-93
		DE-U- 8717822	20-12-90
		DE-U- 8717827	27-09-90
		EP-A, B 0247566	02-12-87
		EP-A, B 0379264	25-07-90
		EP-A- 0494098	08-07-92
		IE-B- 60410	13-07-94
		JP-A- 63000042	05-01-88
US-A-4680060	14-07-87	NONE	
WO-A-9117236	14-11-91	DE-A- 4014747	14-11-91
		AT-T- 116683	15-01-95
		DE-D- 59104162	16-02-95
		EP-A- 0527799	24-02-93
		ES-T- 2066439	01-03-95