PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : B65H 63/06, D01F 2/00	A1	 (11) International Publication Number: WO 94/27902 (43) International Publication Date: 8 December 1994 (08.12.94)
(21) International Application Number: PCT/GBS (22) International Filing Date: 20 May 1994 (2) (30) Priority Data: 08/066,473 24 May 1993 (24.05.93)	20.05.9	BY, CA, CH, CN, CZ, DE, DE (Utility model), DK, ES, FI
 71) Applicant: COURTAULDS FIBRES (HOLDINGS) L [GB/GB]; 50 George Street, London W1A 2BB (Country Close, Goxhi Humberside DN19 7NN (GB). HAYHURST, I John; 90 Bridgeacre Gardens, Coventry CV3 2NP 74) Agent: NEWBY, John, Ross; J.Y. & G.W. Johnson, House, 14-18 High Holborn, London WC1V 6DE 	GB). ill, Sou Malcoli (GB). Furniv	With international search report. h
House, 14-16 High Holooth, London Well V ODE	(OB).	

(54) Title: MANUFACTURE OF SOLVENT-SPUN CELLULOSE FIBRE AND QUALITY CONTROL DETECTION MEANS THERE-FOR

(57) Abstract

Detection means are provided in a method of making continuous filaments of solvent-spun cellulose to detect "trash" or other undesired material on the filaments. Accordingly, there is provided a method of making a tow of continuous filaments of solvent-spun cellulose in which: i) cellulose is dissolved in an amine oxide solvent to form a hot cellulose solution, ii) the hot cellulose solution is extruded through a die assembly to form a tow of continuous filaments, and iii) the tow is passed through a water bath to leach out the amine oxide, wherein the improvement comprises: iv) passing the tow of continuous filaments through detection means, v) the detection means comprising a beam projected across the tow and having receiving means on the opposite side of the tow, the receiving means being calibrated to initiate a signal if obscurement of the beam by the tow varies beyond a predetermined amount. An apparatus including the detection means is also provided.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

ΑT	Austria	GB	United Kingdom	MR	Mauritania
ΑU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	Œ	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PΤ	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgystan	RÜ	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic	SD	Sudan
CG	Congo		of Korea	SE	Sweden
CH	Switzerland	KR	Republic of Korea	SI	Slovenia
CI	Côte d'Ivoire	KZ	Kazakhstan	SK	Slovakia
CM	Cameroon	LI	Liechtenstein	SN	Senegal
CN	China	LK	Sri Lanka	TD	Chad
CS	Czechoslovakia	LU	Luxembourg	TG	Togo
CZ	Czech Republic	LV	Latvia	TJ	Tajikistan
DE	Germany	MC	Monaco	TT	Trinidad and Tobago
DK	Denmark	MD	Republic of Moldova	UA	Ukraine
ES	Spain	MG	Madagascar	US	United States of America
FI	Finland	ML	Mali	UZ	Uzbekistan
FR	France	MN	Mongolia	VN	Vict Nam
GA	Gabon		-		

- 1 -

MANUFACTURE OF SOLVENT-SPUN CELLULOSE FIBRE AND QUALITY CONTROL DETECTION MEANS THEREFOR

This invention relates to the manufacture of cellulose fibre by a method comprising the spinning of continuous 5 cellulose filaments from a solution of cellulose in an organic solvent, particularly an amine oxide solvent. Cellulose manufactured in this manner is known as lyocell and will hereafter be referred to as solvent-spun cellulose or lyocell. The invention particularly aims to provide a detection means 10 to enable the presence of so-called "trash" on the formed continuous filaments to be detected at an appropriate stage in the manufacturing process.

The manufacture of lyocell cellulose filaments is described, for example, in U.S. Patent No. 4,416,698 the contents of which are incorporated herein by way of reference. This Patent discloses a method of producing cellulose filaments by dissolving the cellulose in a suitable solvent such as a tertiary amine N-oxide.

A hot solution of the cellulose is extruded or spun through a suitable die assembly including a jet to produce filamentary material which is passed into water to leach out the amine oxide solvent from the extruded filaments.

The production of artificially formed filaments of material by extruding or spinning a solution or liquid through a spinnerette to form the filaments is, of course, well known. Initially, relatively small numbers of individual filaments were prepared, which filaments were individually wound up for use as continuous filament material. This meant that the number of continuous filaments which needed to be produced was essentially dictated by the number of filaments which could be individually wound either before or after drying.

However, if fibre is produced as a tow or if fibre is produced as a staple fibre then different criteria apply to

the number of filaments which can be produced at any one time. A tow essentially comprises a bundle of essentially parallel filaments which are not handled individually. Staple fibre essentially comprises a mass of short lengths of fibre. Staple fibre can be produced by the cutting of dry tow or it can be produced by forming a tow, cutting it whilst still wet, and drying the cut mass of staple fibre.

Because there is no need to handle individual filaments in the case of a tow product or a staple product, large 10 numbers of filaments can be produced simultaneously.

One problem encountered in the commercial production of solvent-spun cellulose filamentary tows as described above is that "trash" can become attached to the filaments and so degrade their quality. "Trash" in this process is usually in the form of globules of cellulosic polymer formed from the "dope" or hot solution of cellulose. These globules have not been spun into filamentary form and they attach themselves to the filaments of the formed tow. "Trash" can also be formed by pieces of broken filamentary fibre. It can occur from time to time for a variety of reasons. For example, breakage may occur due to the tensions applied to the formed filaments at various points in the manufacturing process. Polymer globules may be caused, for example, by partial blockage of one or more of the spinning holes. Broken filaments, being undrawn, are

Accordingly the present invention seeks to provide detection means in the manufacturing process to alert to the formation of "trash" on the tow of cellulose filaments.

In one aspect, therefore, the invention provides a method 30 of making a tow of continuous filaments of solvent-spun cellulose in which:

- cellulose is dissolved in an amine oxide solvent to form a hot cellulose solution,
- ii) the hot cellulose solution is extruded through a

5

10

30

- 3 -

die assembly to form a tow of continuous filaments,

- iii) the tow is passed through a water bath to leach out the amine oxide, and
- iv) the tow is passed through detection means in which
 - v) a beam is projected across the tow towards receiving means on the opposite side of the tow, the receiving means being calibrated to initiate a signal if obscurement of the beam by the tow varies beyond a predetermined amount.

In another aspect the invention provides an apparatus for the detection of faults on a filamentary tow, which apparatus comprises means to provide a tow of filaments of solvent-spun cellulose, means to transport the tow along a path and detection means positioned on the path, the detection means comprising means to project a beam across the tow and receiving means on the opposite side of the tow, the receiving means being calibrated to initiate a signal if obscurement of the beam by the tow varies beyond a predetermined amount.

The solvent will preferably be a tertiary amine N-oxide and the bath a water bath to leach out the solvent.

The detection means may be installed at any desired position in the manufacturing process and, indeed, detection means may be installed at more than one position in the 25 process, if so desired.

Particularly suitable positions to locate the detection means are:

- i) between the spinning, i.e. extrusion, stage where the filamentary tow is formed and the washing stage to leach out the solvent,
- ii) after the washing stage, and
- iii) if the washed filamentary tow is to have crimp applied to it, immediately before the crimping

- 4 -

stage.

In the latter instance, the tow of fibres, which is normally dried in an oven after the washing stage, will pass through the detection means between the drying stage and the 5 crimping stage.

The detection means preferably comprises a source of collimated infra red light or a laser beam, which is projected across the path of travel of the tow and is received by a photo-receiver, for example a silicon photo diode. The 10 detection means is calibrated so that the desired amount of beam blockage by the desired thickness of the tow causes no alarm signal. However, any change, e.g. increased blockage of the beam caused by "trash" or undesired change in thickness of the tow, causes a change in the electrical output of the 15 photo-receiver. Any change beyond a predetermined amount triggers an appropriate signal. For example, it may trigger an audible alarm.

The detection means is preferably coupled to a microprocessor which has been programmed to analyse the data 20 fed to it by the receiver. The microprocessor can, therefore, initiate any desired alarm and can also be used to maintain overall records for quality control analysis purposes.

It will be appreciated that in a largely automated manufacturing process an audible alarm signal will be 25 desirable in view of the unpredictable and intermittent nature of the occurrence of "trash" on the tow.

Specific embodiments of the invention will now be described by way of example only with reference to the accompanying drawings in which:

Figure 1 is a diagrammatic representation of the various stages in the manufacture of a continuous tow of solvent-spun cellulose fibres, i.e. lyocell;

- 5 -

Figure 2 is a diagrammatic side view showing a detection means positioned in the manufacturing process of Figure 1;

Figure 3 is a plan view of the position shown in Figure 2;

Figure 4 is a side view showing the tow passing through the detector in a first embodiment;

Figure 5 is a similar side view to Figure 4 but showing the tow passing through the beam in a second embodiment; and

Figure 6 is a diagrammatic representation showing the 10 detection means at a different position in the manufacturing process.

In Figure 1, is shown a mixer 10 with inlets 11 and 12 to receive shredded cellulose and an amine oxide solvent respectively. The hot solution is pumped via metering pump 15 13 to a spinnerette 14 where the solution is spun into a continuous tow 15 of fibres.

As the hot tow leaves the spinnerette 14 it is passed through a spin bath 16 in which a mixture of water and the amine oxide is recirculated. At start-up there will be no 20 amine oxide in the spin bath but its proportion to water may rise to about 40% by weight, e.g. 25% by weight. From spin bath 16 the tow is passed via roll 17 through a water bath 18. The tow passing through the water bath may be, for example, up to 12 to 14 inches wide. In the water bath, the amine oxide is dissolved out of the fibres and the tow 19 emerging from the water bath is of solvent-spun cellulose, i.e. lyocell.

From water bath 18 the tow 19 is passed through a nip between rolls 20 and 21 to a detection means 30, which is 30 described in greater detail below with reference to Figures 2 and 3. The tow is then passed through a finishing stage

- 6 -

30A where the filaments are lubricated using spin finishes well known in the art. The tow is then passed through a drying oven 22 maintained at a temperature of about 100 to 180°C e.g. 165°C.

The drying oven is preferably of the perforated drum type, well-known in the art, but may, alternatively, be of the can or calender drier type.

There may be, as shown, a single tow emerging from the spinnerette and this may contain, for example, up to 400,000 10 filaments and may weigh, for example 65 ktex, i.e. 65g/metre, after the drying stage. Alternatively, the spinnerette may produce more than one, for example, four streams of tow and these may contain over 1 million filaments each and weigh, for example, about 181 ktex each after drying.

A single tow passing through the water bath may be, as indicated above, up to 12 to 14 inches (30 to 35 cms) wide. However, where four tows, for example, are produced from the spinnerette, these may be combined into two tows, each pair of tows going through a separate water bath which is at least 20 48 inches (120 cms) wide and each pair of tows 24 inches (60 cms) wide.

The dry tow from drier 22 is then passed into a nip defined by rolls 23 and 24 from which it is fed into stuffer box 25. The crimped tow 26 emerging from the stuffer box is 25 passed via roll 27 to a cutter 28 where it is cut to staple fibre lengths. The crimped staple fibre lengths are collected in box 29.

In Figure 2 and 3 the tow 19 is shown passing from rolls 20 and 21 through the detection means 30. The detection means 30 comprises a counter base 31 above which are set rolls 32 and 33 over which the tow passes. Between rolls 31 and 32 the tow passes in front of an infra-red light source 34 which projects an infra-red beam across the path of travel of the tow. On

- 7 -

the other side of the tow from light source 34 is a silicon photo-diode receiver 35 which detects the infra-red beam passing across the tow. As indicated above, at this point, the tow may be from 12 to 14 inches wide so that light source 5 34 and receiver 35 are spaced apart by a little more than that amount.

As shown in Figure 4, the beam 36 is set so that tow 19 passes centrally through it. If desired the bottom half of the beam, i.e. below tow 19 may be blocked off from the 10 receiver by, e.g. a brass shield 37. This enables the system to run without alarm or counting of occasional loose edges beneath tow.

Alternatively, the beam 36 may be set a little higher relative to the tow 19 so that the tow passes through its 15 lower half - see Figure 5. The tow may be, for example about 2mm above the bottom of a beam of diameter about 10mm. In this set up sensitivity of the detection means is increased towards larger obstructions. In Figure 4, an obstruction of height 2x is seen to produce less than double the obscurement 20 of the beam caused by an obstruction of height x. In Figure 5, an obstruction of height 2x gives obscurement of the beam more than double that caused by an obstruction of height x. Thus it can be appreciated that the position of the beam relative to the tow can be adjusted according to the type and 25 size of trash or other obstruction preferably wished to be detected.

The system is calibrated so that a predetermined level of obscurement of the beam will increment a counter in counter base 31 and sound an alarm (not shown). Counter base 31 may contain or be connected to a microprocessor which may control the alarm and analyse the counter data.

The detection means may also be calibrated to allow for gradual changes in tow thickness whereby it slowly automatically compensates for changes in the amounts of light

- 8 -

received by the receiver. Thus, if for example, 50% of the beam becomes obscured for any length of time, the remaining 50% becomes the "normal" level and the sensitivity is, therefore, doubled. In other words, the detection means counts sudden changes in the level of light received, while at the same time slowly adjusting the notional normal or "zero" obscurement level.

It will be appreciated that various embodiments may be changed from those described above without departing from the scope and spirit of the invention., In particular, the detection means may as previously suggested, be positioned at a different stage in the process. Thus it may be found useful to position a detection means immediately after the filament spinning stage and before the washing stage to leach out the amine oxide solvent. The tow may be positioned to travel just outside the path of the beam so that the beam is only interfered with by, e.g. trash, extending from the tow.

The detection means may, of course, be incorporated in a process in which the tow is not crimped or in which the tow 20 is crimped but is not passed to a cutter.

An alternative embodiment is, therefore, described with reference to Figure 6. In this embodiment a detection means 40 of the same type as described above is positioned between the spinnerette 14 in which the tow of filaments has been 25 formed and the water bath (not shown) in which the solvent is leached out.

In this embodiment the detection means 40 is so positioned that as tow 19 passes over roll 41, the lowermost 30 portion of the beam traverses the travel path of the tow a small distance above the top of the tow. This distance may be adjusted according to the minimum upstanding size of trash or other unwanted material that it is desired to detect. For example, the gap between the beam and the tow may be up to 35 3/8inch (9.5 mm).

- 9 -

The receiving diode in this embodiment may be connected to an alarm, counting means and microprocessor as previously described.

5

10

15

- 10 -

CLAIMS

1. A method of making a tow of continuous filaments of solvent-spun cellulose characterised in that:

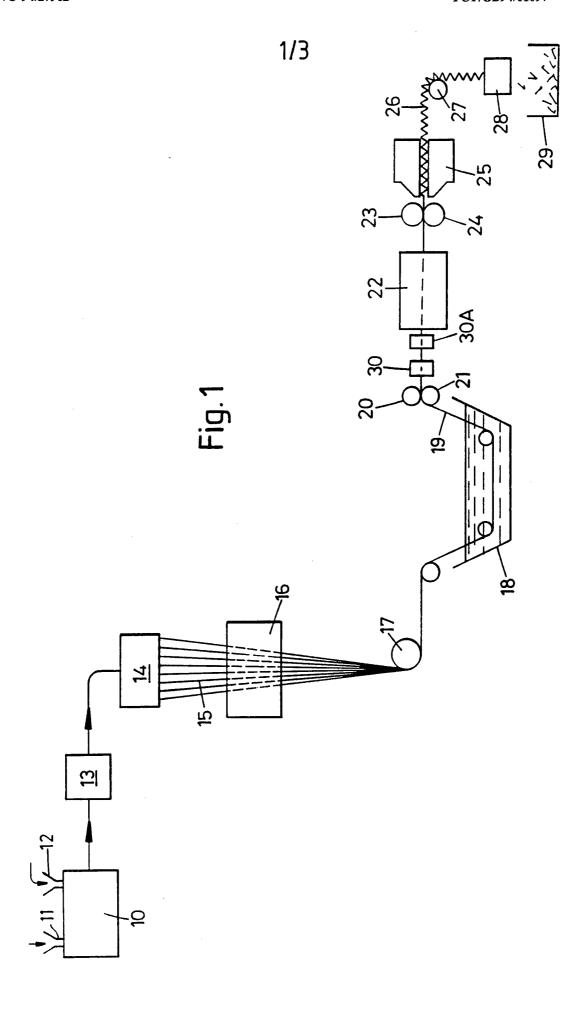
- i) cellulose is dissolved in an amine oxide solvent to form a hot cellulose solution,
- ii) the hot cellulose solution is extruded through a die assembly (14) to form a tow (15) of continuous filaments.
- iii) the tow (15) is passed through a water bath (18) to leach out the amine oxide, and
- iv) the tow (19) is passed through detection means (30) in which a beam (36) is projected across the tow (19) towards receiving means (35) on the opposite side of the tow, the receiving means (35) being calibrated to initiate a signal if obscurement of the beam (36) by the tow varies beyond a predetermined amount.
- A method according to Claim 1, characterised in that the tow (19) is passed through the detection means (30) after
 passing through the water bath (18) and is then dried and, optionally, crimped.
- 3. A method according to Claim 1 or 2, characterised in that the receiving means (35) initiates an audible signal when the beam (36) is obscured beyond said predetermined 25 amount.
 - 4. A method according to Claim 1, 2 or 3, characterised in that the receiving means (35) is coupled to a microprocessor (31) programmed to analyse the data fed to it by the receiver.
- 30 5. An apparatus for the detection of faults on a filamentary tow, characterised in that the apparatus comprises means to provide a tow (19) of filaments of solvent-spun cellulose, means to transport the tow (19) along a path and

detection means (30) positioned on the path, the detection means (30) comprising means (34) to project a beam (36) across the tow (19) and receiving means (35) on the opposite side of the tow (19), the receiving means (35) being calibrated to initiate a signal if obscurement of the beam by the tow (19) varies beyond a predetermined amount.

- 6. An apparatus according to Claim 5, characterised in that it includes means (10) to mix cellulose and solvent to form a hot cellulose solution, means (14) to form a tow (15) 10 of continuous filaments from the hot solution and a bath (18) through which the tow (15) can be passed to leach the solvent from the filaments.
- 7. An apparatus according to Claim 6, characterised in that it includes drying means (22) to dry the tow (19) after 15 it has passed through the bath (18) to leach out the solvent and crimping means (25) to crimp the dry tow (19), the detection means (30) being positioned between the drying means (22) and the crimping means (25).
- 8. An apparatus according to Claim 5, 6 or 7, 20 characterised in that the signal is an audible signal.
 - 9. An apparatus according to any one of Claims 5 to 8, characterised in that the detection means (30) comprises a collimated light source and a photo receiver between which the tow (19) passes.
- 25 10. An apparatus according to any one of Claims 5 to 9, characterised in that the light source is an infra-red light source (34) and the photo receiver is a silicon photo diode receiver (35).
- 11. An apparatus according to any one of Claims 5 to 10, 30 characterised in that the detection means (30) is positioned to project the beam (36) such that the tow (19) passes through the beam (36).

- 12 -

12. An apparatus according to any one of Claims 5 to 10, characterised in that the detection means (40) is positioned to project the beam (36) such that the tow (19) passes just outside the beam (36).



,

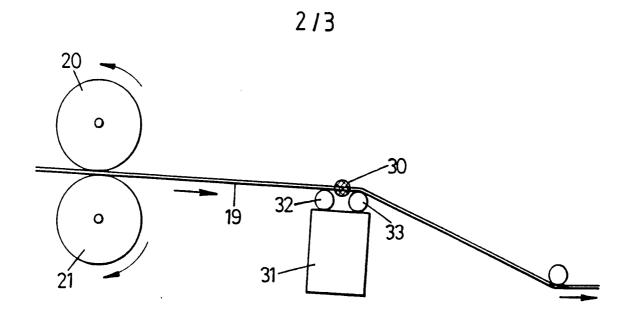


Fig. 2

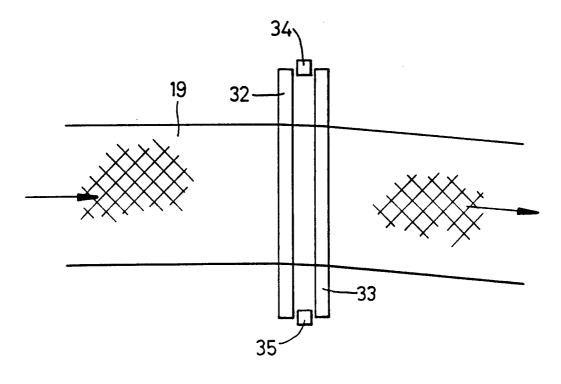


Fig. 3

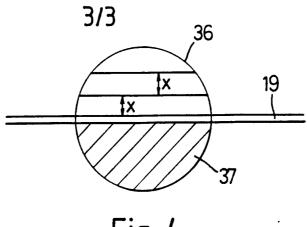
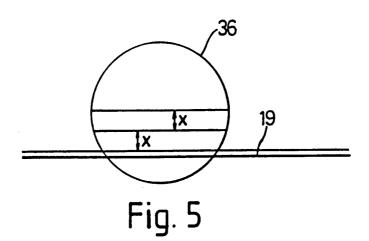
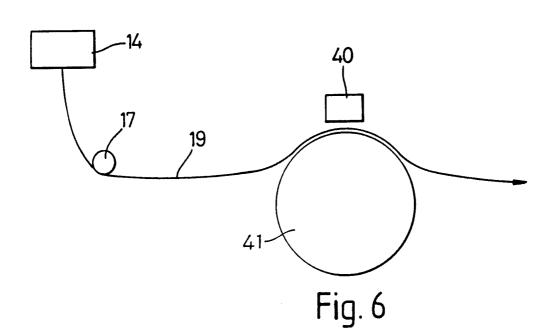


Fig. 4





INTERNATIONAL SEARCH REPORT

Internation pplication No PCT/GB 94/01097

A. CLASS IPC 5	IFICATION OF SUBJECT MATTER B65H63/06 D01F2/00		
According t	to International Patent Classification (IPC) or to both national classi	fication and IPC	
	S SEARCHED		
Minimum d IPC 5	ocumentation searched (classification system followed by classification B65H D01F	ion symbols)	
Documenta	tion searched other than minimum documentation to the extent that	such documents are included in the fields so	arched
Electronic	data base consulted during the international search (name of data ba	se and, where practical, search terms used)	
C. DOCUM	MENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the r	elevant passages	Relevant to claim No.
X	US,A,3 174 046 (HOWARD C. LINDEM AL.) 16 March 1965 see the whole document	ANN ET	1-12
X	US,A,3 447 213 (SIEME DOST ET AL 1969 see the whole document	.) 3 June	1-12
A	US,A,4 416 698 (CLARENCE C. MCCO November 1983 cited in the application	RSLEY) 22	
Fur	ther documents are listed in the continuation of box C.	X Patent family members are listed	in annex.
"A" documeonsi "E" earlier filing "L" docume which citatie	nent which may throw doubts on priority claim(s) or h is cited to establish the publication date of another on or other special reason (as specified)	 "T" later document published after the into or priority date and not in conflict we cited to understand the principle or to invention "X" document of particular relevance; the cannot be considered novel or cannot involve an inventive step when the document of particular relevance; the cannot be considered to involve an indocument is combined with one or not be considered to involve an indocument is combined with one or not be considered. 	ith the application but heory underlying the claimed invention t be considered to ocument is taken alone claimed invention here to be considered to ocupant is taken alone considered invention here to be considered to consider such docu-
'P' docum	ment referring to an oral disclosure, use, exhibition or means nears the published prior to the international filing date but	ments, such combination being obvious in the art. *&* document member of the same paten	ous to a person skilled
	than the priority date claimed e actual completion of the international search	Date of mailing of the international s	
	13 September 1994	2 2. 09. 94	
Name and	mailing address of the ISA European Patent Office, P.B. 5818 Patentiaan 2	Authorized officer	
	NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+ 31-70) 340-3016	Tarrida Torrell,	J

INTERNATIONAL SEARCH REPORT

Information on patent family members

Internation pplication No
PCT/GB 94/01097

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JS-A-3174046		NONE	
JS-A-3447213	03-06-69	NL-A- 6700367	26-02-68
JS-A-4416698	22-11-83	US-A- 4246221	20-01-81
		US-A- 4144080	13-03-79
		AT-B- 387792	10-03-89
		AU-A- 4593779	04-09-80
		BE-A- 875323	04-10-79
		CA-A- 1141913	01-03-83
		DE-A,C 2913589	11-09-80
		FR-A,B 2450293	26-09-80
		GB-A,B 2043525	08-10-80
		JP-C- 1308043	13-03-86
		JP-A- 55118928	12-09-80
		JP-B- 60028848	06-07-85
		NL-A- 7902782	04-09-80
		SE-B- 444191	24-03-86
		SE-A- 7902733	03-09-80
		AT-B- 364900	25-11-81
		AU-A- 3814778	24-01-80
		BE-A- 868737	03-11-78
		CA-A- 1116808	26-01-82
4		DE-A,B,C 2830685	15-02-79
		FR-A,B 2398774	23-02-79
		GB-A,B 2001320	31-01-79
		JP-C- 1048126	28-05-81
		JP-A- 54024963	24-02-79
		JP-B- 55041693	25-10-80
		LU-A- 79932	07-12-78
		NL-A- 7807421	30-01-79
	q	SE-B- 445926	28-07-86
		SE-A- 7808039	27-01-79
		AT-B- 365663	10-02-82
		AU-A- 4066878	17-04-80
		BE-A- 871428	20-04-79
		CA-A- 1135918	23-11-82
		DE-A,B,C 2844163	03-05-79
		FR-A- 2407280	25-05-79
		GB-A,B 2007147	16-05-79

INTERNATIONAL SEARCH REPORT

Information on patent family members

Internation pplication No
PCT/GB 94/01097

Patent document cited in search report	Publication date				
US-A-4416698		JP-C- JP-A- JP-B- NL-A- SE-B- SE-A-	1159007 54073919 57049656 7810788 451856 7811241	25-07-83 13-06-79 23-10-82 02-05-79 02-11-87 01-05-79	