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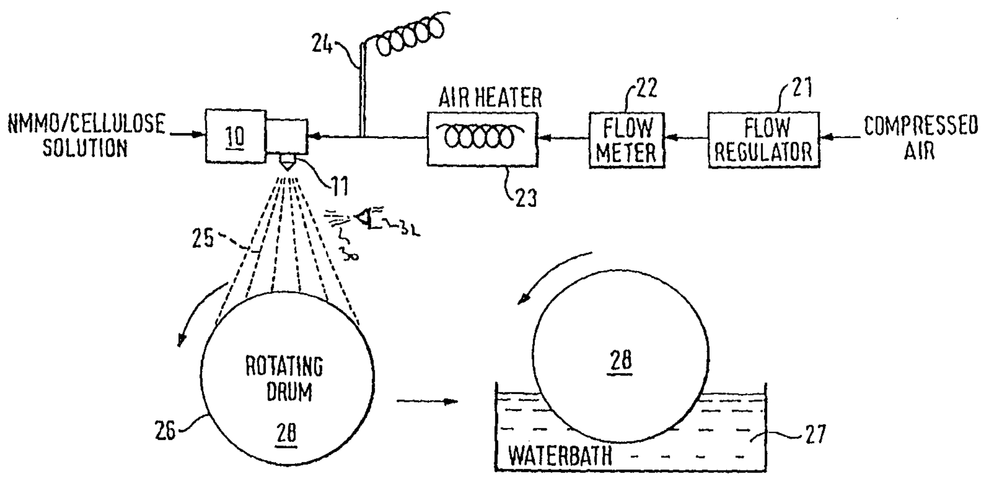
(54) Title: METHODS OF MANUFACTURE OF NONWOVEN FABRIC



(57) Abstract

A method of manufacture of a nonwoven cellulose fabric involves the fibres being formed by extruding a solution of cellulose through at least one spinning jet and subjecting the extrudate fibre to a high velocity gas flow. The fibre passes through a vapour mist which at least partially coagulates the fibre prior to collection as a fibre web. By use of such method, a fabric having a high loft and a relatively low density may be obtained.

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METHODS OF MANUFACTURE OF NONWOVEN FABRIC

This invention relates to a method of manufacture of a nonwoven fabric made from cellulose and in particular from a solution of cellulose.

Cellulose fibres and filaments may be produced by spinning a solution of cellulose in an amine oxide solvent which is then leached into water or a dilute solution of aqueous amine oxide, to produce cellulose filaments which can then be cut into staple fibres. The process of extrusion and coagulation is referred to as solvent spinning, and the fibres of solvent-spun cellulose so produced are known under the generic name of lyocell.

It is possible to produce small decitex fibres below 1.0 dtex by disintegrating staple fibres. However, this is costly, and requires a high energy consumption.

The present applicant's international patent application PCT/GB97/03391 discloses a method of manufacture of a nonwoven cellulose fibre made from fibres formed by extruding a solution of cellulose through at least one spinning jet and subjecting the extrudate fibre to a high velocity gas flow, the fibres being collected on a surface on which the fibre is subsequently coagulated. The fibres tend to bond together before coagulation forming a relatively dense nonwoven fabric which is highly bonded.

In some applications a highly bonded, high density fabric web is not desirable, for example for filtration materials, or for materials which require a high water holding capacity.

The present invention provides a method of manufacture of a nonwoven cellulose fabric having a high loft and a relatively low density.

According to the present invention there is provided a method of manufacture of a non-woven cellulose fabric in which extrudate fibre produced from a spinning jet is subjected to a high velocity gas flow, and the fibre is passed through a vapour mist which at least partially coagulates the fibre prior to its collection as a fibre web.

The extruded fibre is attenuated by the gas flow and may be broken down into indefinite lengths.

The vapour mist may be formed from any suitable coagulant. The mist is preferably an aqueous mist, although lower alkyl alcohols, for example, may also be used. Conveniently, atomised water may be sprayed from a nozzle located below the spinning jet.

The cellulose solution is preferably a solution of cellulose in an amine oxide solvent, typically a tertiary N-amine oxide and in particular N-methylmorpholene-N-oxide (NMMO). The cellulose solution may contain 4-22% by weight of cellulose, preferably 10-15%, having a degree of polymerisation of 200-5,000, and more typically 400-1,000.

In a preferred embodiment the cellulose solution comprises 14% by weight of cellulose, 12% by weight water and 74% by weight of NMMO, the cellulose having a degree of polymerisation of about 600.

The attenuated fibre-forming microfibres or fibrils are collected and are then further coagulated (alternatively referred to as being "regenerated") by means of water, or a dilute aqueous solution of amine oxide containing up to 20% amine oxide in water. The fibrils may be collected directly into a coagulation bath, or may be collected onto a surface and then further coagulated.

The gas, preferably air or dry steam, is blown onto the extruded fibres at a velocity of between 200 $m.s^{-1}$ (metres per second) and 500 $m.s^{-1}$ and has a temperature of between 100°C and 155°C, preferably about 145°C for a cellulose concentration of 14-15%. The lower the cellulose content of the dope, the lower the air temperature that can be used. The gas velocity should be at least 50 times higher than the velocity of the extrudate fibre emerging from the spinning jet, and preferably between 1,000 and 20,000 times said velocity. The air is directed onto the fibre extrudate at a bias angle, preferably of between 15° and 45° relative to the longitudinal axis of the extrudate, and more preferably about 30°. The air jets may also be biased at a second skew angle relative to the spinning jet so that the air jet axes and fibre axis do not intersect, the air jets being tangential to the surface of the fibre extrudate.

The fibre collecting surface may be flexible, or resilient. Preferably the fibre collection is made onto a bed of foam which is preferably soaked in water or aqueous NMMO solution.

The invention also provides a nonwoven lyocell fabric in which the fibres are bonded or entangled together without the use of a binder, the fabric having a density no greater than 175 $g.dm^{-3}$, and/or a tensile extension to break of at least 7%.

The invention will hereinafter be described in more detail by way of example only, with reference to the accompanying drawings in which:-

Figure 1 is a schematic drawing of an embodiment of apparatus for the production of a nonwoven fabric according to the present invention;

Figure 2 is a plan view of a spinning jet nozzle used in the apparatus of Figure 1;

Figure 3 is a side elevation of the nozzle shown in Figure 2, with internal passages ghosted; and

Figure 4 is an axial cross-section through the nozzle shown in Figure 2 and Figure 3.

With reference to Figure 1, there is shown an extruder 10 having a nozzle 11 attached thereto. The extruder is fed with a solution comprising 14% by weight cellulose, 12% by weight of water and 74% by weight of N-methylmorpholene-N-oxide (NMMO). The cellulose has an average degree of polymerisation of about 600.

The cellulose solution may be manufactured as is described in WO 94/28217. The cellulose solution in the extruder is held at a temperature of between 95 and 110°C, preferably 105°C, and is forced through the nozzle to extrude as a continuous filament of cellulose dope.

The nozzle 11 is shown in Figures 2 and 3 and may be secured directly onto the extruder 10, or may be secured to an adapter (not shown) which in turn is secured to the extruder 10. The nozzle 11 has a hollow screw threaded stud 13 on its back face 12 and a central passageway 14 which terminates in a jet aperture 15. The jet has a diameter of between 0.2 and 0.3mm, and preferably about 0.27mm.

The cellulose dope is forced into the passageway 14 under pressure, and is extruded through the jet 15. The nozzle 11 also has a plurality of gas outlet passageways 16, preferably three, spaced around the central passageway 14. Each gas passageway 16 is inclined with respect to the jet axis, and they are circumferentially equally spaced around the jet 15 so that each gas stream exiting its respective passageway 16 has the same effect upon the extrudate filament.

The gas passageways 16 make a bias or convergence angle with the longitudinal axis of the jet of between 15° and 45°, and more preferably 30°. The passageways 16 are also skewed so that the axes of the passageways 16 do not themselves converge. The gas passageways are about 2.0 mm in diameter.

The back face 12 of the nozzle has an annular groove 17 therein which interconnects the ends of the three passageways 16.

When the nozzle is secured to the extruder, the central passageway 14 is connected to the cellulose dope feed and the annular passageway 17 is connected to a gas supply, preferably compressed air.

With reference to Figure 1, compressed air is fed from a source (not shown) through a flow regulator valve 21, a flow meter 22, a heater 23 and a temperature sensor 24, to the air passageway 17 in the nozzle. The sensor 24 may be connected to the air heater 23 for control of the air temperature.

The extrudate filaments emerging from the nozzle 11 are subjected to attenuation by high velocity air streams 25 emerging from the outlet passageways 16, and the filament is drawn and may be fractured and either blown directly into a coagulation bath 27 or, preferably, blown onto a support surface 26 situated about 30 cm from the nozzle 11. The cellulose dope flow rate is preferably about 0.2 $g.min^{-1}$. In the illustrated embodiment the support surface 26 is formed by the outer peripheral surface of a rotatable drum 28, which turns at about 10 revolutions per minute (rpm) to build up a layer of nonwoven fabric on the drum.

The extrudate filaments pass through a fine mist 30 of water vapour, generated by water sprayed from an air atomising nozzle 32 located about 10 cm below nozzle 11.

The surface of the drum may be covered in compliant material such as a layer of foam, preferably a 2mm thick layer of polyurethane foam having an average cell size of 50μm. The foam may be soaked in water or an aqueous solution of NMMO.

Subsequent to the formation of the nonwoven fabric layer on the drum 28, the drum 28 is immersed in a coagulant bath

27 containing a suitable coagulant such as water or a dilute solution of amine oxide in water, to coagulate the nonwoven cellulose fabric on the drum. The fabric layer is then dried on the drum after washing to remove solvent.

Table 1 below summarises the various conditions used in the formation of extruded filament.

Table 1

Sample reference	Method of laydown	Air temperature (°C)	Air flowrate ($l.s^{-1}$)	Mean fibre diameter (μm)
1	Collected dry	145	2.5	7
2	Collected on wet surface	148	2.3	5
3	" + water aerosol	147	2.3	5
4	" " + compliant surface	145	2.3	7

An air flow rate of 2.4 $l.s^{-1}$ (litres/second) corresponds approximately to air velocity of 250 $m.s^{-1}$.

Samples 3 and 4 were made in accordance with present inventions.

Table 2 below summarises web loftiness by way of thickness measurements, carried out using a Mitutoyo Dial Thickness Gauge, fitted with film/web measurement plates. Because basis weights are different, the thickness is normalised to a basis weight of 25 $g.m^{-2}$.

Table 2

Sample reference	Basis Weight ($g.m^{-2}$)	Web Thickness (μm)	Normalised Thickness ($\mu m/25g.m^{-2}$)	density ($g.dm^{-3}$)
1	25.3	113	112	223
2	28.3	145	128	195
3	28.0	168	150	166
4	22.5	163	181	138

It can be seen that the samples according to the invention produce less dense fabrics having a density no greater than 170 $g.dm^{-3}$.

With reference to Sample 4, it is believed that the foam serves two purposes, namely (a) nascent fibres striking a hard collector surface tend to fuse on impact - a compliant surface removes some of the impact energy and reduces bonding and (b) on compression of the foam at impact, coagulant contained in its pores is expressed to the surface and aids rapid regeneration.

To assess mechanical properties, strips were cut from the webs, 5 mm in width, and tested on a Testometric PCX materials tensile tester at a gauge length of 20 mm and at cross head speed of 20 mm/min. Along with the absolute tensile strengths, the tensile strengths are also shown normalised to a basis weight of the web of 25 $g.m^{-2}$, which better reflects the comparative mechanical properties, as basis weight variations are eliminated.

Table 3 summarises the properties of the fabric webs formed on the drum 26.

<u>Table 3</u>

Sample reference	Tensile strength (N) normalised to 25 $g.m^{-2}$	Elongation @ break (mm)	Comments
1	3.7	0.7	Clean breaks
2	0.2	0.85	Fairly clean breaks
3	0.41	1.54	Sample maintained integrity after "breaking"
4	0.09	2.5	Sample maintained integrity after "breaking"

For samples 2, 3 and 4, where the surface of the drum is partially immersed in the coagulation bath so that the drum surface is wet, further coagulation takes place on contact with the wet drum or previously laid fibres.

For sample 1, the surface of the drum is dry and the fabric is regenerated after build up on the drum.

Samples 3 and 4, collected on the drum after passing through the vapour mist, clearly had lower tensile strength but greater extension to break whilst still maintaining some integrity after the break point. These high loft material are suitable for use as filters and high absorbency materials.

Whilst the invention has been described for the attenuation of cellulose dope containing 14% cellulose, it can be applied to other dopes as is described in PCT/GB97/03391, the contents of which are hereby incorporated into the present application by way of reference.

Claims

1. A method of manufacture of a nonwoven cellulose fabric made from fibres formed by extruding a solution of cellulose through at least one spinning jet and subjecting the extrudate fibre to a high velocity gas flow, the fibre passing through a vapour mist which at least partially coagulates the fibre prior to collection as a fibre web.

2. A method as claimed in claim 1, wherein the cellulose solution is a solution in an amine oxide solvent, and the vapour mist is an aqueous mist.

3. A method as claimed in claim 1 or claim 2, wherein the gas flow rate is at least 200 meters per second.

4. A method as claimed in claim 3, wherein the gas flow is at least 50 times faster than the flow rate of the extrudate.

5. A method as claimed in any one of claims 1 to 4, wherein the gas of the gas flow has a temperature of at least 100°C.

6. A method as claimed in any one of claims 1 to 5, wherein the mist is formed by an atomiser nozzle which is spaced from and below the spinning jet.

7. A method as claimed in any one of claims 1 to 6, wherein the cellulose solution contains from 4-22% by weight cellulose, preferably about 10 to 15% by weight of cellulose.

8. A method as claimed in any one of claims 1 to 7, wherein the cellulose has an average degree of polymerisation of about 600.

9. A method as claimed in any one of claims 1 to 8, wherein said gas flow comprises compressed air which is

directed onto the fibres at a bias angle of about 30°C to the axis of the extrudate fibre.

10. A method as claimed in any one of claims 1 to 9, wherein the fibre web is collected onto a dry surface and the web is subsequently further treated with coagulant.

11. A method as claimed in any one of claims 1 to 9, wherein the fibre web is collected on a surface which is wetted by coagulant.

12. A method as claimed in any one of claims 1 to 11, wherein the fibre web is collected on a compliant surface.

13. A method as claimed in claim 12, wherein the compliant surface is provided by a foam layer about 2 mm in thickness.

14. A nonwoven lyocell fabric made by a method as claimed in any one of claims 1 to 13.

15. A nonwoven lyocell fabric web in which fibres are bonded together without the use of a binder, the fabric web having a density of less than 175 $g.dm^{-3}$.

16. A nonwoven fabric as claimed in claim 14, wherein the fabric has an elongation to break of at least 7%.

17. A nonwoven lyocell fabric web in which the fibres are bonded together without the use of a binder, where the fabric web has an elongation to break of at least 7%.

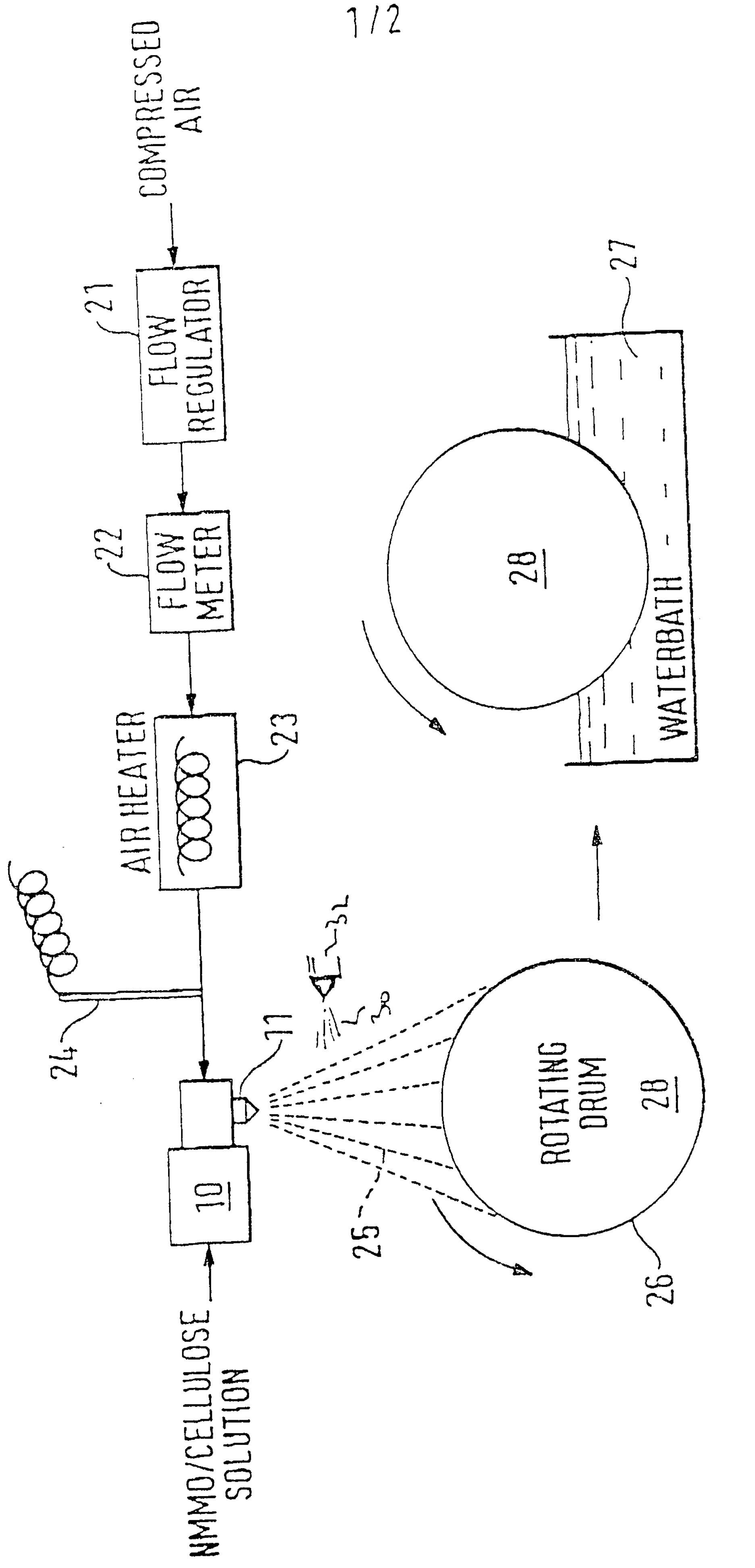
COMPRESSED AIR
FLOW REGULATOR
21
FLOW METER
22
AIR HEATER
23
24
11
10
NMMO/CELLULOSE SOLUTION
25
ROTATING DRUM
28
26
27
WATERBATH
28

FIG. 1

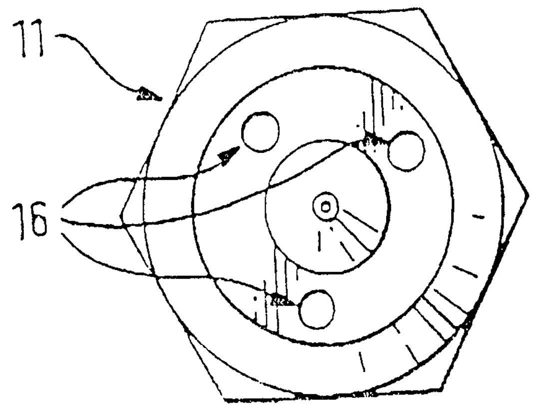

FIG. 2

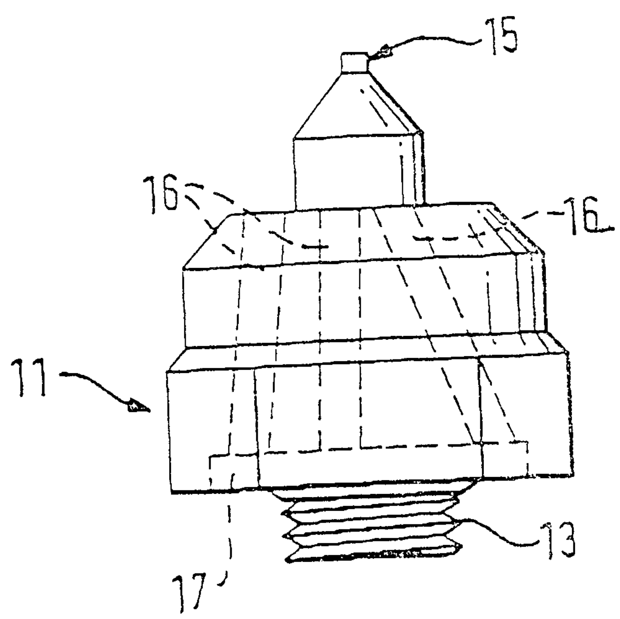

FIG. 3

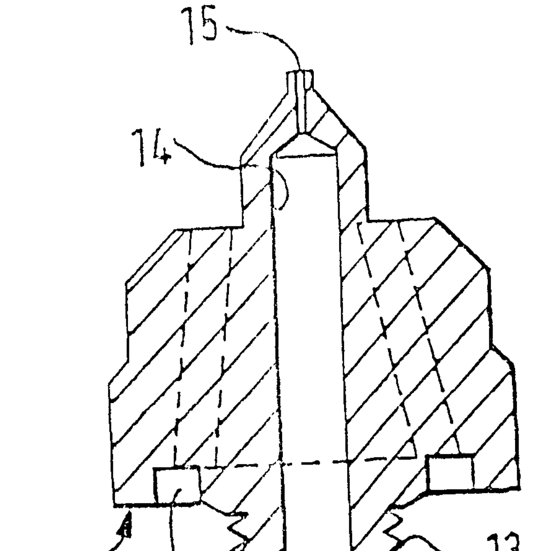

FIG. 4

INTERNATIONAL SEARCH REPORT

Inter· ·onal Application No
PCT/GB 99/01775

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D01F2/00 D01D5/098 D04H1/56 D04H3/16

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 D01F D01D B01D D04H

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

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Y	US 5 601 771 A (RUEF HARTMUT) 11 February 1997 (1997-02-11) examples ---	1,5,10, 14
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
9 September 1999	27/09/1999

Name and mailing address of the ISA	Authorized officer
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Inter 'onal Application No
PC1/GB 99/01775

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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