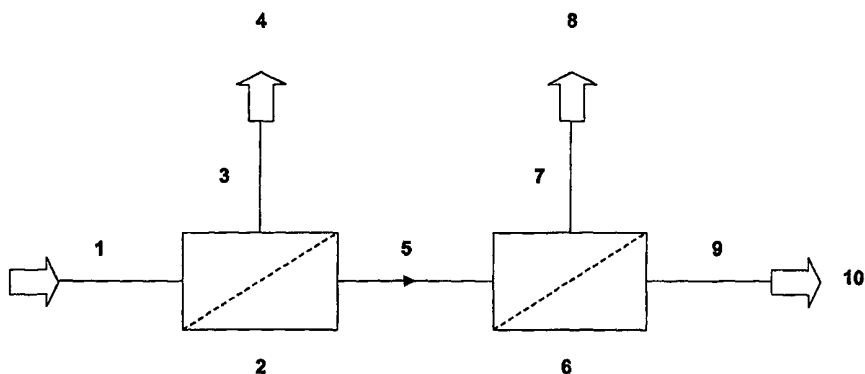




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(21) International Application Number: PCT/EP98/04422 (22) International Filing Date: 16 July 1998 (16.07.98) (30) Priority Data: 97112200.7 17 July 1997 (17.07.97) EP (71) Applicant (for all designated States except US): PPG INDUSTRIES LACKE GMBH [DE/DE]; Stackenbergstrasse 34, D-42329 Wuppertal (DE). (72) Inventors; and (75) Inventors/Applicants (for US only): KASCHA, Dietmar [DE/DE]; Im Eich 14, D-65760 Eschborn 2 (DE). MANDERSCHIED, Karl [DE/DE]; Mommsenstrasse 30, D-50935 Köln (DE). LINDEMANN, Johannes [DE/DE]; Heerstrasse 6, D-53619 Rheinbreitbach (DE). (74) Agent: STERNAGEL & FLEISCHER; Braunsberger Feld 29, D-51429 Bergisch Gladbach (DE).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published With international search report.	

(54) Title: METHOD OF TREATING SOLVENT-CONTAINING PROCESS LIQUIDS



(57) Abstract

The present invention relates to a method of obtaining a solvent-containing concentrate from used rinsing liquids from the paint processing industry which comprise solids fractions, organic solvents and water by: a) separating the rinsing liquid (1) by ultrafiltration (2) into a retentate (3) and a permeate (5), b) separating the ultrafiltration permeate (5) by reverse osmosis (6) into a solvent-containing concentrate (7) and into a permeate (9) which is substantially free of organic solvents, and c) separately removing (4, 8, 10) the ultrafiltration retentate (3), the solvent-containing concentrate (7) from the reverse osmosis (6), and the reverse osmosis permeate (9).

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**Method of treating solvent-containing
process liquids**

The present invention relates to a method of
5 obtaining a solvent-containing concentrate from used
rinsing liquids from the paint processing industry.

The initial rinsing liquids which are used to
rinse and clean the paint supply in painting units as a
consequence of colour changeover, change of coating
10 material, cleaning, maintenance, etc., contain organic
solvents in an amount of 10-50% by weight in deionized
water. In the course of the cleaning process the paint
supply apparatuses are flushed with deionized water.
Used rinsing liquids, which are obtained in painting
15 processes in the automotive industry when waterborne
paint systems are used, therefore have in general a
solids content of 4 - 15% by weight and a proportion of
low molecular mass organic solvents of up to 15% by
weight, the remainder being deionized water, based on
20 the overall mass of the rinsing liquid.

The concepts known and employed to date for the
treatment of used rinsing liquid include incineration,
distillation and direct introduction of the rinsing
liquid into the circuit water of spray booths for the
25 painting process. Because of the high water content of
the rinsing liquid, the energy consumption entailed by
the first two concepts set out above is very high. In
addition, the emissions of environmental pollutants are
unacceptably high in the case of incineration and of
30 introduction into the circuit water, and additional
costs and hazards arise from the transportation of the
rinsing liquid from its site of formation to the site
of disposal, which is generally necessary for incinera-
tion and distillation. In the case of incineration and
35 of introduction into the circuit water, moreover, valu-
able substances, such as organic solvents and deionized
water, are lost.

There is therefore a need to provide a more
cost-effective and environmentally compatible treatment

method for solvent-containing process liquids from the paint processing industry.

EP-A-0 675 080 discloses a method of concentrating the overspray produced by wet washing-out in spray booths by means of a multistage membrane filtration. In this method, the booth water/overspray mixture to be concentrated is initially concentrated in an ultrafiltration stage by separating it into a retentate and a permeate, which contains low molecular mass constituents. The ultrafiltration permeate is supplied together with a nanofiltration permeate to a reverse osmosis stage. The retentate of the reverse osmosis and the retentate of the ultrafiltration are supplied to a nanofiltration stage, where separation takes place into a retentate and a permeate. In terms of its concentration and paint composition, the retentate of the nanofiltration corresponds substantially to the aqueous paint employed and can therefore be used for this purpose directly or as a constituent of a paint composition. The permeate of the reverse osmosis can be recycled into the booth water circuit.

The key point of this method is that even the low molecular mass constituents of the paint, such as crosslinking agents, are separated off and recycled into the concentrated retentate of the ultrafiltration. With this method, ultimately, the concentration of the paint constituents of the booth water/overspray mixture takes place with separation of water. As a result, this method is incapable of recovering the fractions of organic solvent in the rinsing liquid.

The object on which the present invention is based is therefore to provide a cost-effective treatment method for solvent-containing rinsing liquids which makes it possible, in particular, to use both the solvents and the process water again.

This object is achieved by a method of obtaining a solvent-containing concentrate from used rinsing process liquids from the paint processing

industry, comprising solids fractions, organic solvents and water, by:

- 5 a. separating the rinsing liquid by ultrafiltration into a retentate and a permeate,
- b. separating the ultrafiltration permeate by reverse osmosis into a solvent-containing concentrate and into a permeate which is substantially free of
10 organic solvents, and
- c. separately removing the ultrafiltration retentate, the solvent-containing concentrate from the reverse osmosis, and the reverse osmosis permeate.
15

The ultrafiltration of the present invention is a membrane separation process which is carried out with membranes having a cutoff point of from 200 to 500,000 Da, preferably from 1000 to 250,000 Da. The pressure
20 difference between entry and exit of the module in the ultrafiltration of the invention is 0.5 - 5 bar, preferably below 1 bar. With this mode of operation it is possible to achieve a permeate flow of from 10 to 80 l/m²h, preferably from 15 to 50 l/m²h. In this con-
25 text, the permeate flow of the ultrafiltration depends not only on this pressure difference but also, in particular, on the feed temperature. The transmembrane pressure difference in the ultrafiltration of the invention is between 1 - 5 bar.

30 All customary commercial membrane modules are suitable for the ultrafiltration unit employed in the method of the invention, such as cushion modules, plate modules, spiral modules, tubular modules, capillary modules, multi-channel modules or hollow-fibre modules.
35 The membranes can be made of polysulphone, polyacrylonitrile, polyolefins such as polypropylene or polyethylene, Teflon, porous carbon, ceramic (such as e.g. silicon carbide and alumina), cellulose acetate, polyurea, aromatic or aliphatic polyamides, sulphonated

polyaryl ethers, polyfuran, polybenzimidazole, various fluoropolymers, such as polyvinylidene fluoride, polyether-aromatic compounds, such as polyimide or polyimidazopyrrolidone, or similar materials. The person skilled in the relevant art knows how to select the appropriate ultrafiltration unit for the separation task at hand. Modules which have been found to be particularly suitable for the ultrafiltration stage of the invention are multi-channel modules or tubular modules with alumina, silicon carbide, polyvinylidene fluoride and polyacrylonitrile membranes.

Using an ultrafiltration stage as described above, a process liquid from the paint processing industry can be separated into a concentrate and into a permeate which essentially contains only constituents having a molecular weight of less than 1000 Da, preferably less than 200 Da.

In a particularly preferred embodiment of the present invention the membrane used for the ultrafiltration stage has a large pore size, corresponding to a cutoff point of 250,000 - 500,000 Da, and is doped with charge carriers, preferably positive charge carriers. A membrane of this kind achieves both a very sharp separation and a very high permeate flow at low differential pressures, between the entry and exit of the module, of less than 1 bar. Surprisingly, it has been found for this membrane that both the permeate flow and the separation performance are greater at low differential pressures, between entry and exit of the module, of less than 1 bar, than at higher pressures, so that particularly effective separation at especially favourable cost is possible with such a membrane.

In order to achieve the desired separation result, the ultrafiltration can also be practised in a plurality of stages.

The reverse osmosis of the invention is likewise a membrane separation method, using membranes which retain at least 95% by weight, preferably at least 98% by weight and, more preferably, at least 99%

by weight of sodium chloride. The transmembrane pressure difference is normally between 15 and 100 bar, preferably between 20 and 60 bar and, more preferably, between 20 and 50 bar. In this case a permeate flow of
5 from 3 to 30 l/m²h is achieved.

Customary membrane modules are suitable for the reverse osmosis, such as spiral modules, tubular modules, capillary modules and hollow-fibre modules. When selecting the membrane materials particular care should
10 be taken to ensure that the membranes are resistant to the organic solvents, especially aprotic organic solvents, which become concentrated in the retentate of the reverse osmosis. In this context, suitable materials are those described above in connection with the
15 ultrafiltration. Polyamide membranes have been found to be particularly suitable.

Using a reverse osmosis stage of this kind, according to the invention, it is possible to obtain a permeate which is essentially free of organic solvents.
20 For the purposes of the invention "essentially free" means a concentration of organic solvents in the reverse osmosis permeate of less than 0.2% by weight based on the overall mass of the permeate, preferably of less than 0.1% by weight, more preferably of less
25 than 0.05% by weight and, most preferably, of less than 0.01% by weight. The separation can be improved further, if required, by a multistage reverse osmosis method.

In another preferred embodiment of the method
30 of the invention it is possible following the reverse osmosis stage to provide a pervaporation stage as well. By pervaporation is meant a diffusion-controlled membrane separation technique in which a vapour mixture which becomes established above a mixture of liquids is
35 separated as a result of the differing permeability of an appropriate membrane. Continual pumped removal of the vapour on the gas side of the membrane produces a concentration gradient which maintains the diffusion. Pervaporation is particularly suitable for concentrat-

ing further the retentate of the reverse osmosis. Alternatively and/or additionally it can be used to remove traces of solvent in the reverse osmosis permeate, or may totally replace the reverse osmosis stage
5 in the method of the invention.

Further preferred embodiments of the method according to the invention are defined in the subclaims.

The method of the invention is particularly
10 suitable for treating used rinsing liquids which are obtained when cleaning and rinsing the paint supply of painting units in the automotive industry. This relates in particular to painting units which process waterborne paint systems. Rinsing liquids which have
15 been found suitable for such painting units comprise, in addition to deionized water, one or more solvents selected from isobutanol, n-butanol, n-propanol, isopropanol, ethylene glycol monobutyl ether, butyldiglycol and N-methylpyrrolidone.

20 The used rinsing liquids obtained in such operations generally have a solids content of 4 - 15% by weight, a proportion of low molecular mass organic solvents of up to 15% by weight, the remainder being deionized water, based on the overall mass of the rinsing liquid. The solids fraction is composed essentially
25 of the components of the particular paints used. These components comprise pigments and binders in the range between 2000 and 250,000 Da, and low molecular mass constituents, especially crosslinking agents, which may
30 have a molecular weight of less than 2000 Da. The organic solvents originate mainly from the rinsing liquid and to a small extent from the particular paint system used, or are elimination products of the paint system.

35 The principle of the method of the invention is illustrated in Figure 1.

Key:

(1) used rinsing liquid

- 7 -

- (2) ultrafiltration stage
- (3) retentate of the ultrafiltration stage
- (4) re-use of the retentate of the ultrafiltration stage
- 5 (5) permeate of the ultrafiltration stage
- (6) reverse osmosis stage
- (7) retentate of the reverse osmosis stage
- (8) re-use of the retentate of the reverse osmosis stage
- 10 (9) permeate of the reverse osmosis stage
- (10) re-use of the permeate of the reverse osmosis stage

The used rinsing liquid 1 is collected in a
15 tank (not shown) and then passed into an ultrafiltration stage 2 for reprocessing. In the ultrafiltration stage, the high molecular mass constituents of the rinsing liquid are concentrated in the retentate 3 to a solids content of preferably at least 50% by weight.
20 The retentate 3 of the ultrafiltration is then supplied for re-use 4 - for example, for direct use as paint constituents. The permeate 5 from the ultrafiltration stage 2 is essentially free from constituents of relatively high molecular mass, with a molecular weight of
25 more than 1000 Da, preferably more than 2000 Da, and contains organic solvents and water. This permeate 5 is supplied to a reverse osmosis stage 6 in which the organic solvents in the retentate 7 are concentrated to a concentration of at least 25% by weight, preferably
30 at least 50% by weight, and are supplied for separate use 8 - for example, as initial rinsing liquid. The permeate 9 of the reverse osmosis 6 is essentially pure water and can, for example, be recycled into the rinsing process or else employed for other applications
35 where deionized water is required.

The present invention is illustrated further by the following example:

A used rinsing liquid having the composition stated in Table 1 was passed from a reservoir, at a

reservoir temperature of 20°C, through an ultrafiltration stage for concentration. In the ultrafiltration stage, a tubular module with a ceramic membrane having a cutoff point of 100,000 daltons was employed. The
5 rinsing liquid entered the module on the feed side at 3 m³/h, the pressure upstream of the module being 2.3 bar and the pressure downstream of the module being 2.0 bar. Ultrafiltration was conducted to a solids content of 50% by weight in the concentrate. The composition
10 tion of the permeate and of the retentate of the ultrafiltration was determined analytically and is shown in Table 1.

The permeate of this ultrafiltration was then supplied to a reverse osmosis stage. In the reverse
15 osmosis stage, a spiral module with a polyamide membrane having a sodium chloride retention of not more than 99.2% was used. The ultrafiltration permeate entered on the feed side at 0.75 m³/h, the transmembrane pressure difference being 41 bar. The reverse osmosis
20 was conducted up to a solvent concentration of 25% in the concentrate. The composition of the concentrate and of the permeate of the reverse osmosis was determined analytically and is reproduced in Table 1.

Table 1

Description	Ultrafiltration				Reverse osmosis			
	Original	Permeate	Conc.	Retention	Feed	Permeate	Conc.	Retention
Concentration, %		62	62			65	65	
Membrane		SiC	SiC			SWCl	SWCl	
Methanol	0.08	0.07	0.10	12.50	0.07	0.02	0.16	71.43
n-Propanol	2.25	2.20	2.33	2.22	2.20		6.29	100.00
i-Butanol	0.17	0.15	0.20	11.76	0.15		0.43	100.00
n-Butanol	0.14	0.10	0.21	30.00	0.10		0.29	100.00
Butylglycol	5.97	4.83	7.83	19.10	4.83		13.80	100.00
Butyldiglycol	0.95	0.70	1.36	26.32	0.70		2.00	100.00
DMEA	0.08	0.07	0.11	16.00	0.07		0.20	100.00
N-Methylpyrrolidone	2.35	2.30	2.43	2.13	2.30		6.57	100.00
Total solvents	12.00	10.42	14.57	13.14	10.42	0.02	29.73	99.81
Water	86.14	89.58	85.43		87.40	99.98	67.50	
Unknown	1.86							
Total	100.00	100.00	100.00		100.00	100.00	100.00	
Solids	13.08	0.9	17.00		0.90	<0.1	0.96	
Volatiles	86.92	99.1	83.0		99.10	>99.9	99.04	

CLAIMS

1. Method of obtaining a solvent-containing concentrate from used rinsing liquids from the paint processing industry which comprise solids fractions, organic solvents and water by:
- 5
- a. separating the rinsing liquid (1) by ultrafiltration (2) into a retentate (3) and a permeate (5),
- 10
- b. separating the ultrafiltration permeate (5) by reverse osmosis (6) into a solvent-containing concentrate (7) and into a permeate (9) which is substantially free of organic solvents, and
- 15
- c. separately removing (4, 8, 10) the ultrafiltration retentate (3), the solvent-containing concentrate (7) from the reverse osmosis (6), and the reverse osmosis permeate (9).
- 20
2. Method according to Claim 1, characterized in that the ultrafiltration (2) is practised in a plurality of stages.
3. Method according to either of the preceding claims, characterized in that the ultrafiltration (2)
- 25
- is carried out at a pressure difference between entry and exit of the ultrafiltration membrane of between 0.5 and 5 bar, preferably below 1 bar.
4. Method according to one of the preceding claims, characterized in that the ultrafiltration membrane has a pore size which corresponds to a cutoff point of 250,000 Da - 500,000 Da and is doped with charge carriers.
- 30
5. Method according to Claim 4, characterized in that the ultrafiltration membrane is doped with positive charge carriers.
- 35
6. Method according to either of Claims 4 and 5, characterized in that the ultrafiltration is carried out with a pressure difference between entry and exit of the ultrafiltration membrane of from 0.5 to 1 bar.

7. Method according to one of the preceding claims, characterized in that the membrane used for the reverse osmosis retains at least 95%, preferably at least 98% of sodium chloride.
- 5 8. Method according to Claim 7, characterized in that the reverse osmosis membrane is resistant to aprotic solvents.
9. Method according to one of the preceding claims, characterized in that the permeate of the reverse osmosis (6) is purified further with a pervaporation stage.
- 10 10. Method according to one of the preceding claims, characterized in that the concentrate of the reverse osmosis (6) is concentrated further with a pervaporation stage.
- 15 11. Method according to one of Claims 1-8, characterized in that the reverse osmosis (6) is practised in a plurality of stages.
12. Method according to one of Claims 1-8, characterized in that the reverse osmosis stage (6) is replaced by a pervaporation stage.
- 20 13. Method according to one of the preceding claims, characterized in that the rinsing liquid comprises a mixture of one or more solvents preferably selected from isobutanol, n-butanol, n-propanol, isopropanol, ethylene glycol monobutyl ether, butyldiglycol and N-methylpyrrolidone.
- 25 14. Method according to one of the preceding claims, characterized in that the ultrafiltration permeate (5) is free of particels.
- 30 15. Method according to claim 14, characterized in that the ultrafiltration permeate (5) is vaporizable nearly free from residue.
16. Method according to one of the preceding claims, characterized in that the concentrate of the reverse osmosis (6) and/or the pervaporation stage has a content of organic solvents of at least 25% by weight, preferably of at least 40% by weight, more preferably of at least 45% by weight.
- 35

17. Method according to one of the preceding claims, characterized in that the permeate of the reverse osmosis (6) and/or the pervaporation stage is essentially free of organic solvents.

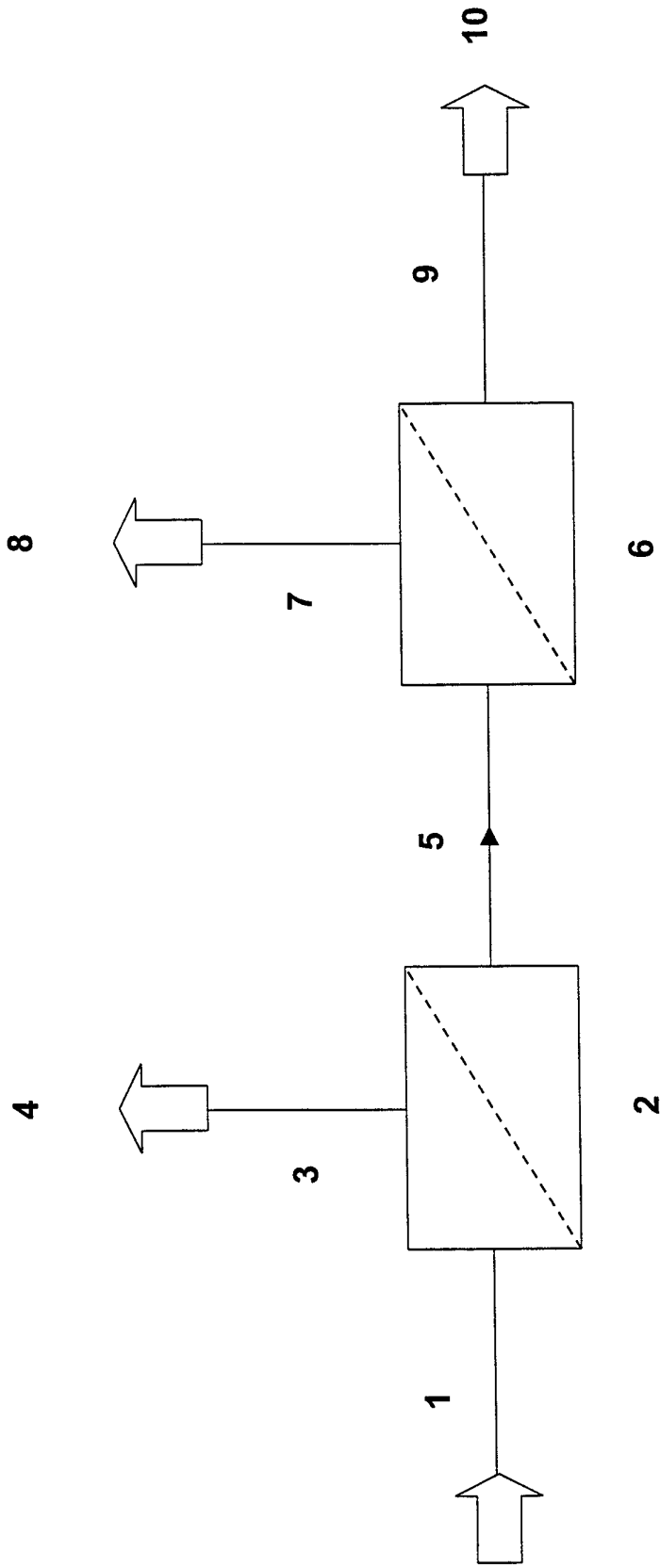


FIGURE 1

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 98/04422

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C02F1/44 B01D61/58 //B05B15/02

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 C02F B01D

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.
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Y	---	3-6,12 11,16
A	EP 0 536 648 A (HERBERTS & CO GMBH) 14 April 1993 see abstract; claims 1,4; figure see column 2, line 20 - line 29 see column 4, line 30 - line 38 see column 6, line 25 - column 7, line 37 see column 9, line 20 - line 40	3
Y	---	1
A	---	1
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

2 November 1998

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 98/04422

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	PATENT ABSTRACTS OF JAPAN vol. 008, no. 130 (C-229), 16 June 1984 & JP 59 041498 A (NITTO DENKI KOGYO KK), 7 March 1984 see abstract -& DATABASE WPI Section Ch, Week 8416 Derwent Publications Ltd., London, GB; Class M11, AN 84-097115 XP002051718 see abstract ---	1,7,8
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