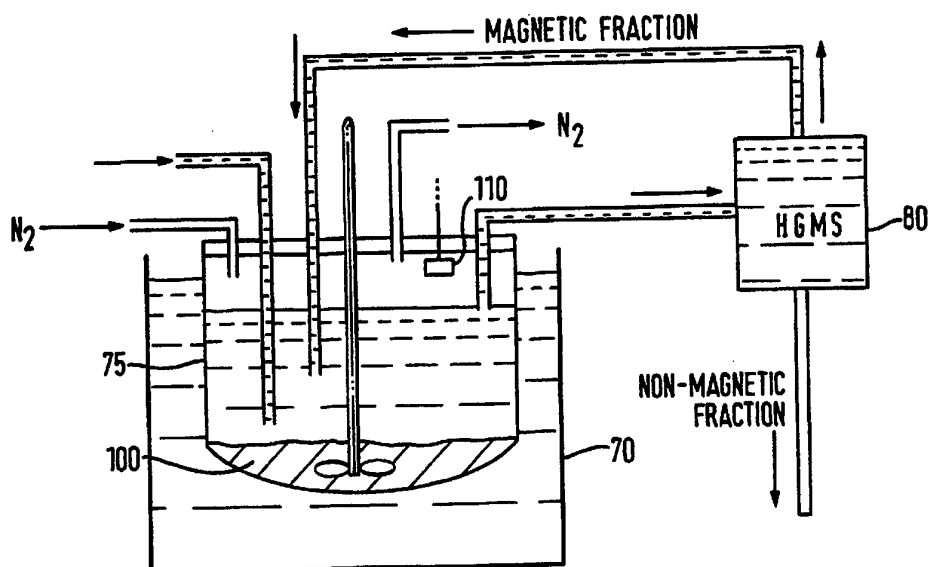




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<p>(21) International Application Number: PCT/GB96/01993 (22) International Filing Date: 15 August 1996 (15.08.96) (30) Priority Data: 9516753.2 16 August 1995 (16.08.95) GB (71) Applicant (for all designated States except US): UNIVERSITY OF SOUTHAMPTON [GB/GB]; Highfield, Southampton SO17 1BJ (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): WATSON, James, Henry, Peter [GB/GB]; 61 Pointout Road, Bassett, Southampton SO16 7DL (GB). ELLWOOD, Derek, Clifford [GB/GB]; Brakenburn, Manesty, Keswick, Cumbria CA12 5UG (GB). (74) Agent: TURNER, James, Arthur; D. Young &amp; Co., 21 New Fetter Lane, London EC4A 1DA (GB).</p>		<p>(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</p>

(54) Title: MAGNETIC SEPARATION



## (57) Abstract

A method of generating an adsorbent product for use in magnetic separation of contaminants from an influent liquid comprises the steps of: (i) mixing one or more types of micro-organism with the contaminated influent liquid in a chemostat vessel; (ii) magnetically separating a magnetic fraction of liquid from the chemostat vessel from a non-magnetic fraction; (iii) returning the magnetic fraction to the chemostat vessel; and (iv) collecting precipitated material from the chemostat vessel for use as the adsorbent product.

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MAGNETIC SEPARATION

This invention relates to magnetic separation.

Magnetic separation is a technique used to remove contaminants such as heavy  
5 metal ions from solution in, for example, water.

One example of the use of magnetic separation is to remove radioactive heavy  
metal contaminants from waste water generated in a nuclear plant. The technique  
involves adding an adsorbent material to the contaminated solution which attaches to  
the contaminants, for example by chemical or electrostatic adsorption. The adsorbent  
10 material has magnetic properties so that, after the adsorbent material has removed  
heavy metals and/or organic materials from solution, the loaded adsorbent can be  
removed magnetically. (However, other separation techniques such as microfiltration,  
high speed centrifuge, hydroclone or flotation could be used).

A complementary process to the above technique is the so-called biomagnetic  
15 separation process. The basis of previously proposed biomagnetic separation  
techniques is that low-level micro-organisms are grown and then introduced into the  
contaminated solution. The micro-organisms have the two important properties  
mentioned above: they interact with the contaminants in the solution (generally by  
precipitation or adsorption on the organism surface) and they have magnetic  
20 properties so that they can subsequently be separated from the solution using a  
magnetic technique such as high gradient magnetic separation (HGMS). When the  
micro-organisms are separated from the solution in this way, they carry with them the  
precipitated contaminants, and so the contaminants are removed from the solution.

This process is described in various publications such as the article  
25 "Biomagnetic Separation And Extraction Process For Heavy Metals From Solution",  
Watson & Ellwood, Minerals Engineering, Vol. 7, No. 8, pp1017-1028 (1994), and  
"A Biomagnetic Separation Process For The Removal Of Heavy Ions From Solution",  
Watson & Ellwood, Proceedings of the International Conference on Control of  
Environmental Problems from Metal Mines, 1988.

30 Figure 1 is a schematic diagram of such a previously proposed biomagnetic  
separation apparatus, comprising a chemostat 10 in which the micro-organisms (in  
this example, the so-called "*Desulfovibrio*" micro-organism) are grown.

The *Desulfovibrio* micro-organisms are then supplied to a reaction vessel 20 in which they are mixed (using a stirrer 30) with contaminated effluent and solutions of sulphates ( $\text{SO}_4$ ) and lactates. In the reaction vessel 20 the heavy metal contaminants in the effluent precipitate onto the surface of the *Desulfovibrio* micro-organisms.

The mixture is then passed to a high gradient magnetic separator 40 which (as described in the published references listed above) comprises a matrix of fine ferromagnetic wire which is magnetised by an externally-applied magnetic field (not shown). The paramagnetic *Desulfovibrio* bacteria (with precipitated contaminants) are attracted and held onto the wires by magnetic forces. The decontaminated effluent then emerges through an outlet 50.

From time to time, the material accumulated on the matrix can be removed by switching off the applied magnetic field and washing the particles from the matrix. Alternatively, the matrix can simply be withdrawn from the magnetic field for washing. Thus, HGMS is a cyclical process with a collection phase and a washing phase.

In the schematic diagram of Figure 1, the *Desulfovibrio* bacteria with the heavy metal contaminants emerge through a separate washing outlet 60 during the washing of the matrix.

A problem with these previous magnetic separation processes is the difficulty in identifying suitable micro-organisms (from a large number of available micro-organisms) or other materials to interact with the contaminants in the particular effluent to be treated and produce a strongly magnetic precipitate.

This invention provides magnetic separation apparatus in which contaminants are separated from an influent liquid by magnetic separation of micro-organisms which combine with or attach to the contaminants, the apparatus comprising:

a chemostat vessel for growing the micro-organisms and for mixing the micro-organisms with the contaminated influent liquid; and

a magnetic separator for receiving liquid from the chemostat vessel and for separating a magnetic fraction of the liquid from a non-magnetic fraction, the magnetic fraction being returned from the magnetic separator to the chemostat vessel.

This invention also provides apparatus for generating an adsorbent product for

use in magnetic separation of contaminants from an influent liquid, the apparatus comprising:

a chemostat vessel for growing the micro-organisms and for mixing the micro-organisms with the contaminated influent liquid; and

5 a magnetic separator for receiving liquid from the chemostat vessel and for separating a magnetic fraction of the liquid from a non-magnetic fraction, the magnetic fraction being returned from the magnetic separator to the chemostat vessel.

This invention also provides a method of generating an adsorbent product for use in magnetic separation of contaminants from an influent liquid, the method comprising the steps of:

10 (i) mixing one or more types of micro-organism with the contaminated influent liquid in a chemostat vessel;

(ii) magnetically separating a magnetic fraction of liquid from the chemostat vessel from a non-magnetic fraction;

15 (iii) returning the magnetic fraction to the chemostat vessel; and

(iv) collecting precipitated material from the chemostat vessel for use as the adsorbent product.

The invention recognises that the problem of selecting suitable micro-organisms for use in treating a particular contaminated liquid can be solved by growing a "cocktail" of a number of different micro-organisms in a chemostat, and then using a *magnetic feedback* process to isolate those which interact with the contaminants to give a magnetically separable product.

20 At the same time, undesired micro-organisms from the cocktail (i.e. those which do not interact with the contaminants to give a magnetic product) can be diverted away from the chemostat, to avoid interference with the remainder of the magnetic separation process. This can dramatically improve the success, and therefore the economic viability, of the magnetic separation process.

25 The operator does not need to worry about **which** micro-organisms of the cocktail are promoted by the feedback process, and which are discarded. This is because the selection is made on the basis of the desired properties of the micro-organisms, so those micro-organisms which are promoted in the feedback chemostat are those which are useful in the separation process for that (or those) contaminant(s)

in the current liquid to be treated. However, if the micro-organisms which are promoted by the feedback process using a sample of effluent are analysed and identified, a similar mixture of micro-organisms could then be sold commercially as a medium for treating that effluent.

5           The skilled man will appreciate that the magnetic separation of the magnetic fraction from the non-magnetic fraction need not be 100% efficient. The intention is that magnetic fraction tends to be returned to the vessel in preference to the non-magnetic fraction.

10           The advantage described above relates to the selection of suitable micro-organisms. However, the method and apparatus of at least embodiments of the invention take matters one stage further, by recognising that the magnetic product generated in the feedback chemostat is itself an adsorbent of the contaminants in the liquid to be treated.

15           In embodiments of the invention, the magnetic feedback chemostat is first used with a selection of micro-organisms. Those which give favourable results, by combining with the contaminant(s) to give a magnetic product, are recycled into the chemostat, while those which do not are discarded. The magnetic product which is returned to the chemostat forms a slurry at the bottom of the chemostat. This slurry tends to be formed of micro-organisms (which may well be dead by this stage) on  
20           which, for example, sulphur products of iron and sulphur products of the contaminants are precipitated. These (generally dead) precipitated micro-organisms are themselves useful as adsorbents of the contaminants, as is the material precipitated on the micro-organisms, even if it becomes detached from the micro-organisms. Accordingly, the slurry (adsorbent material) which collects at the bottom of the  
25           feedback chemostat can be removed and used in the magnetic treatment of further contaminated liquid in a mixing vessel after which the contaminant-loaded adsorbent can be removed magnetically.

30           However, in other embodiments of the invention, if it is known that a particular micro-organism is suitable for use with the current contaminant(s), the techniques described above could still be employed to produce the adsorbent product from that micro-organism.

          Preferably the chemostat vessel comprises an interior vessel supported within

a temperature controlled water bath.

In the method, preferably steps (ii) and (iii) are performed cyclically a plurality of times. Also, preferably step (i) comprises mixing two or more types of micro-organism with the contaminated influent. (i.e. a cocktail is used, so that the method leads to promotion of the most suitable micro-organisms).

An embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings, throughout which like parts are referred to by like references, and in which:

Figure 1 is a schematic diagram of a previously proposed biomagnetic separation apparatus;

Figure 2 is a schematic diagram of a magnetic feedback chemostat; and

Figures 3a and 3b schematically illustrate techniques for recovering an adsorbent slurry from the chemostat of Figure 2.

Referring now to Figure 2, an influent liquid comprising a contaminated solution of heavy metals is supplied at a dilution rate of 0.1 (10%) per hour to a temperature-controlled water bath 70 of the chemostat vessel 75 containing a mixture or cocktail of micro-organisms, iron, sulphates, and a suitable nutrient compound.

An example list of sulphide-generating micro-organisms which could be included in the cocktail is as follows:

*Desulfovibrio*  
*Desulfatomaculum*  
*Desulfomonas*  
*Desulfobulbus*  
*Desulfococcus*  
*Desulfobacterium*  
*Desulfobacter*

In the temperature-controlled water bath, the micro-organism particles multiply. Some of the micro-organisms of the cocktail will tend to attach to the heavy metal contaminants, while others will not attach to the particular contaminants present.

Nitrogen gas is also supplied to assist the multiplication of the micro-organisms.

Liquid is drawn off from the temperature-controlled water bath to a high gradient magnetic separator 80 which separates a magnetic fraction from a non-magnetic fraction. The non-magnetic fraction contains decontaminated liquid and any unwanted micro-organisms (i.e. micro-organisms which do not form a magnetic product with the current contaminants), and is diverted away.

However, during a washing phase of the HGMS 80, the magnetic fraction is returned to the bath 70. This contains the magnetic product formed by interaction of certain of the micro-organisms and the contaminants. (It is not necessary to identify which particular micro-organisms are promoted in this way; the only thing that matters is that they form the magnetic product).

In this way, one or more suitable micro-organisms, which combine with the contaminants to generate a magnetic product, are promoted in the chemostat vessel. Once this population has been identified, the population can be prepared and marketed as a micro-biological product to treat that particular effluent. However, in a further stage, it has been recognised that the magnetic product generated in the feedback chemostat by such a process is itself an adsorbent of the contaminants in the liquid to be treated.

The magnetic product which is returned to the chemostat forms a slurry layer 100 at the bottom of the chemostat. This slurry tends to be formed of micro-organisms (which may well be dead by this stage) on which, for example, sulphur products of iron and sulphur products of the contaminants were precipitated. These (generally dead) micro-organisms are themselves useful as electrostatically or chemically bonded adsorbents of the contaminants, as is the material precipitated on the micro-organisms, even if it becomes detached from the micro-organisms. Accordingly, the slurry which collects at the bottom of the feedback chemostat can be removed and used in the magnetic treatment of further contaminated liquid in a conventional chemostat arrangement, by mixing the adsorbent slurry with the liquid to be decontaminated and then incubating the mixture, typically for several hours.

Various modifications of the basic process described above are envisaged in further embodiments of the invention.

The magnetic susceptibility of the adsorbent can be increased by adding erbium and/or dysprosium ions (as erbium or dysprosium salts such as chlorides or



ethylene diamine tetra acetates (EDTAs)) either during the feedback process described above or at the end of the process when the slurry is recovered.

The example above referred to the production of sulphides of iron. However, other metals such as mercury could be used, and sulphates as well as (or instead of) sulphides could be produced. Furthermore, instead of producing sulphides using the *Desulfovibrio* or other sulphide-generating micro-organism, other products such as phosphates and/or oxides could be produced by using micro-organisms appropriate to those salts such as *Candida Utilis* or *Metallo Reducians* respectively. The performance of the adsorbent slurry produced with these alternative salts can be enhanced by adding erbium and/or dysprosium as described above.

The techniques described above are not only suitable for use in recovering heavy metal contaminants; they can also be used for removing organic contaminants such as chloro- and fluoro-carbon compounds. This is particularly true for adsorbent products based on sulphides.

Although the apparatus described above allows the adsorbent product to be collected as a slurry from the bottom of the vessel, it could instead be collected by techniques such as froth flotation (described in the reference 'Mineral Processing Technology, 3rd Edition, BA Wills, Pergamon Press, 1985); membrane filtering, high speed centrifugal filtering or hydroclone techniques.

Finally, it has been observed that a possible by-product of the process is hydrogen sulphide ( $H_2S$ ) which can be produced if excess sulphate ions are present in the reaction vessel. Hydrogen sulphide can tend to act as a precipitant of the contaminant, but is a much less efficient adsorbent than the iron sulphide products attached to the micro-organisms. It is therefore preferable to reduce the hydrogen sulphide production in order to maximise or at least improve production of the microbiological sulphides.

Hydrogen sulphide production could be reduced by simply adding large excess amounts of iron to the vessel, to eliminate any free sulphur in the vessel. However, to do this in an uncontrolled manner can increase the operating costs of the apparatus (since unnecessary amounts of iron are being added) and can have other disadvantages in that a large excess of iron would affect the molar ration of the iron-sulphur products  $Fe_xS$  which are generated, which in turn can affect the adsorption efficiency.

Therefore, in an embodiment of the invention, the production rate of hydrogen sulphide is monitored by sampling the gas present above the liquid surface using a conventional electronic hydrogen sulphide detection element 110. Iron is then added to the chemostat at a rate which is controlled using conventional feedback techniques (not shown), to aim to keep the hydrogen sulphide production below a threshold amount.

Figures 3a and 3b illustrate two techniques for retrieving the adsorbent material 100 from the vessel 75. In Figure 3a, a dip tube 77 is used in a collection phase to pump the material from the bottom of the vessel 75 (i.e. the material which was deposited earliest). In Figure 3b, a trap-door or similar opening 78 is provided at or near the lowest point of the vessel 75 (with a passageway 79 provided through the water bath 70) to allow the earliest-deposited material to be retrieved.

In summary, embodiments of the invention relate to the production of microbiological populations which, for industrial effluents, can produce magnetic adsorbent material. For different effluents there may be different populations of micro-organisms produced.

CLAIMS

1. Magnetic separation apparatus in which contaminants are separated from an influent liquid by magnetic separation of micro-organisms which combine with or attach to the contaminants, the apparatus comprising:
- 5 a chemostat vessel for growing the micro-organisms and for mixing the micro-organisms with the contaminated influent liquid; and
- a magnetic separator for receiving liquid from the chemostat vessel and for separating a magnetic fraction of the liquid from a non-magnetic fraction, the magnetic fraction being returned from the magnetic separator to the chemostat vessel.
- 10
2. Apparatus for generating an adsorbent product for use in magnetic separation of contaminants from an influent liquid, the apparatus comprising:
- a chemostat vessel for growing the micro-organisms and for mixing the micro-organisms with the contaminated influent liquid; and
- 15 a magnetic separator for receiving liquid from the chemostat vessel and for separating a magnetic fraction of the liquid from a non-magnetic fraction, the magnetic fraction being returned from the magnetic separator to the chemostat vessel.
- 20
3. Apparatus according to claim 1 or claim 2, in which the chemostat vessel comprises an interior vessel supported within a temperature controlled water bath.
4. Apparatus according to any one of claims 1 to 3, in which the micro-organisms comprises one or more micro-organisms selected from the group consisting of:
- 25 sulphide-generating micro-organisms;  
sulphate-generating micro-organisms;  
oxide-generating micro-organisms; and  
phosphate-generating micro-organisms.
- 30
5. Apparatus according to any one of the preceding claims, comprising:  
means for detecting the rate of hydrogen sulphide production within the

chemostat vessel; and

means for adding iron to the chemostat vessel in amounts dependent on the rate of hydrogen sulphide production.

5

6. A method of generating an adsorbent product for use in magnetic separation of contaminants from an influent liquid, the method comprising the steps of:

(i) mixing one or more types of micro-organism with the contaminated influent liquid in a chemostat vessel;

10

(ii) magnetically separating a magnetic fraction of liquid from the chemostat vessel from a non-magnetic fraction;

(iii) returning the magnetic fraction to the chemostat vessel; and

(iv) collecting precipitated material from the chemostat vessel for use as the adsorbent product.

15

7. A method according to claim 6, in which steps (ii) and (iii) are performed cyclically a plurality of times.

20

8. A method according to claim 6 or claim 7, in which step (i) comprises mixing two or more types of micro-organism with the contaminated influent.

25

9. A method according to claim 8, in which the micro-organisms comprises one or more micro-organisms selected from the group consisting of:

sulphide-generating micro-organisms;

sulphate-generating micro-organisms;

oxide-generating micro-organisms; and

phosphate-generating micro-organisms.

30

10. A method according to any one of claims 6 to 9, comprising the step of adding erbium and/or dysprosium ions to the chemostat vessel.

11. A method according to any one of claims 6 to 10, in which step (iv) comprises

collecting a slurry from the bottom of the chemostat vessel.

12. A method according to any one of claims 6 to 11, comprising the steps of:  
monitoring the rate of hydrogen sulphide production in the chemostat vessel;  
5 and  
adding iron to the chemostat vessel in amounts dependent on the rate of  
hydrogen sulphide production.
13. An adsorbent product generated using apparatus according to any one of  
10 claims 1 to 5 and/or a method according to any one of claims 6 to 13.

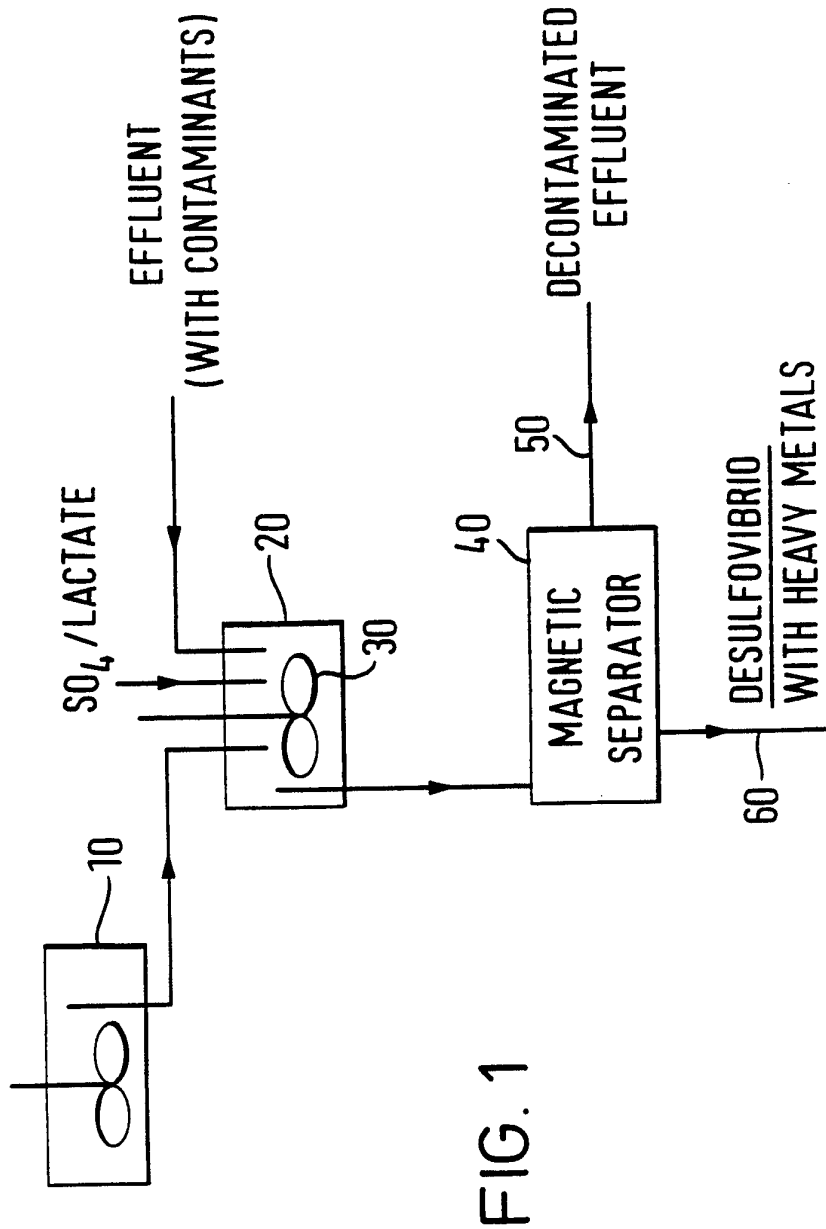
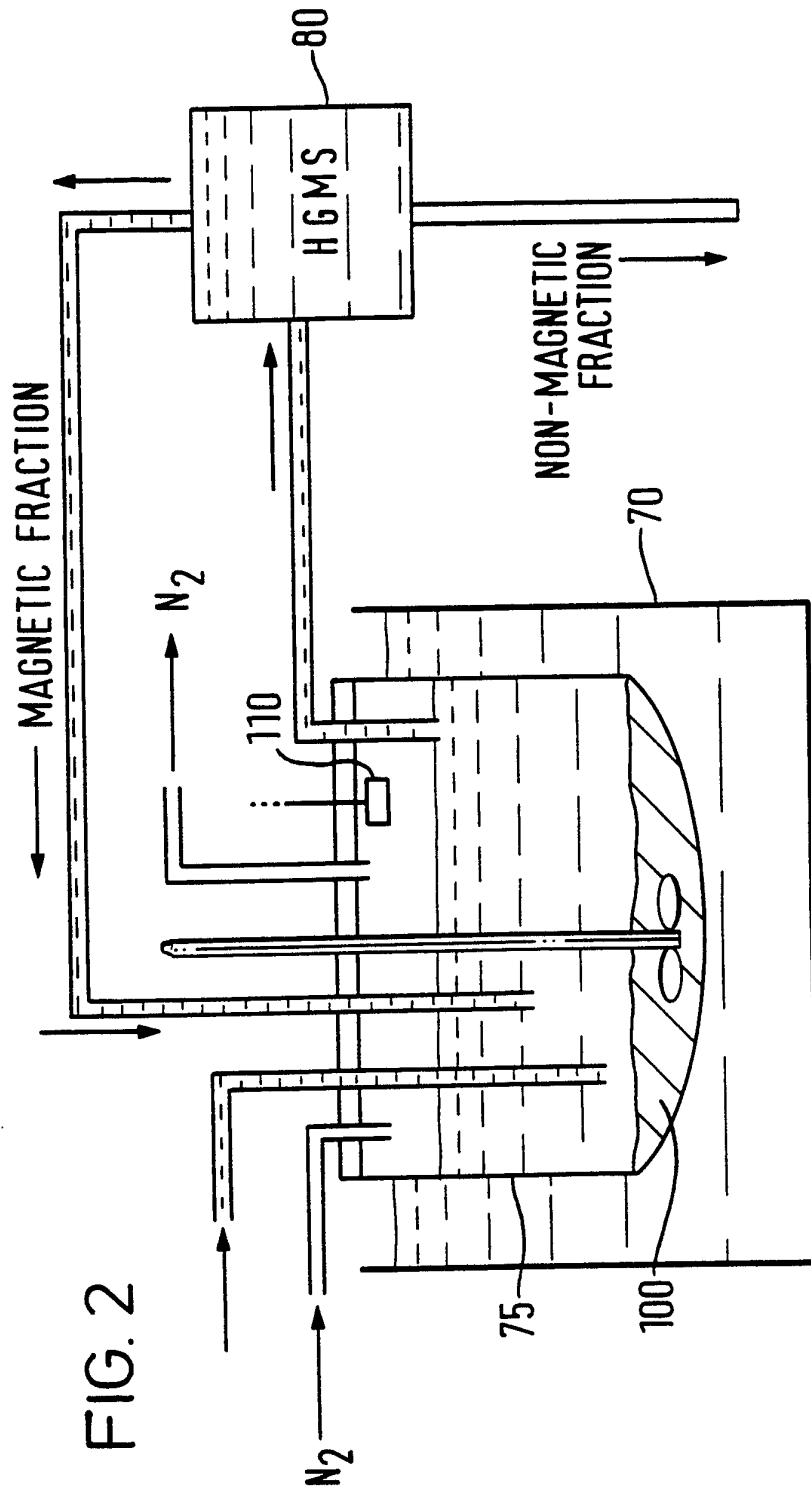


FIG. 1



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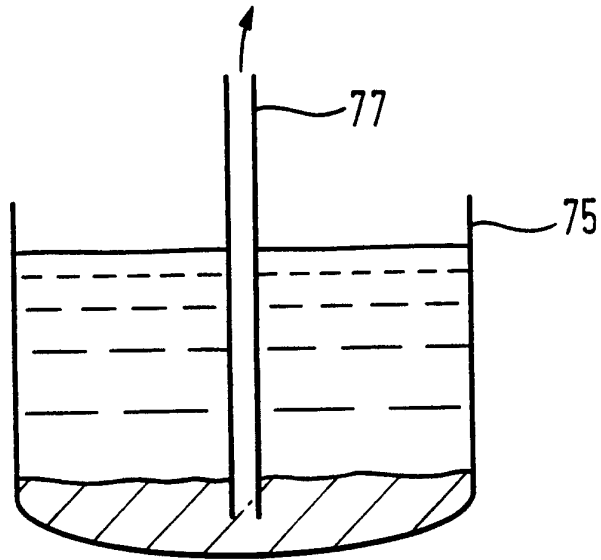


FIG. 3a

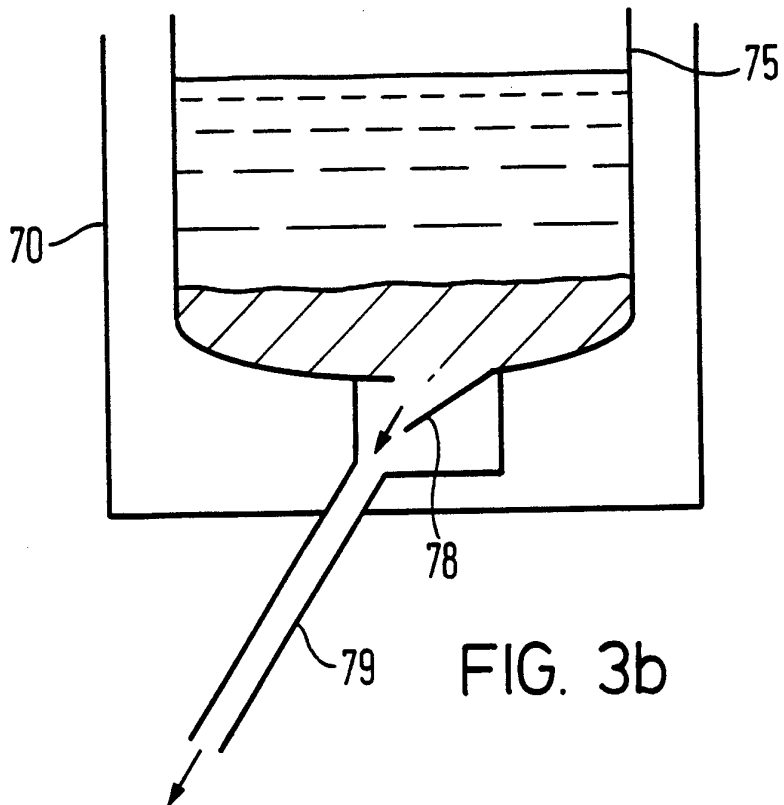


FIG. 3b



# INTERNATIONAL SEARCH REPORT

national Application No  
PCT/GB 96/01993

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 C02F3/34 C02F9/00 C02F1/48

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

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.
X	US 4 735 725 A (REISCHL ARTUR ET AL) 5 April 1988	1,2,4, 6-9,11, 13
Y	see column 2, line 30  see column 2, line 53 - line 64 see column 1, line 27 - line 33; claims; figures see column 3, line 3 - line 30 see column 4, line 18 - line 51 ---	1,4-10, 12
Y	US 5 328 594 A (HETTINGER WILLIAM P) 12 July 1994 see claims ---	10
Y	US 4 508 625 A (GRAHAM MARSHALL D) 2 April 1985 see claims ---	10
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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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- "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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Date of the actual completion of the international search

14 January 1997

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

national Application No PCT/GB 96/01993
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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Information on patent family members

national Application No

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