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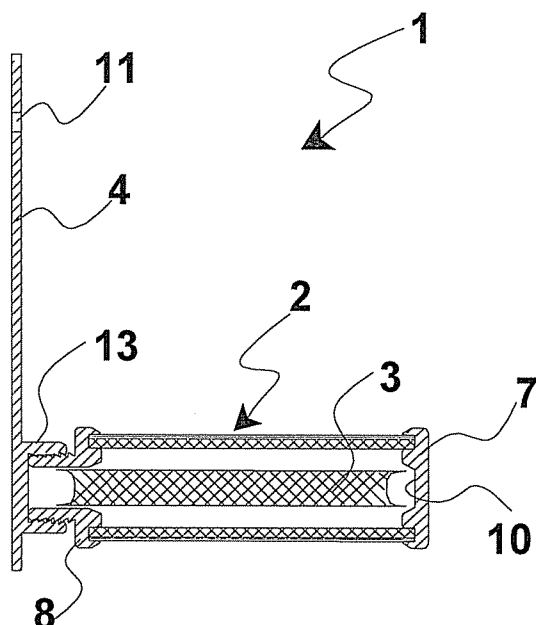
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(54) Title: SAMPLING DEVICE FOR VOLATILE SUBSTANCES



(57) Abstract: Sampling device for volatile polluting substances by radial symmetry diffusion which, in addition to allowing high sampling flow rates, allows to either prevent or considerably reduce the adsorption of some substances, such as for example steam or ozone, potentially interfering with the detection of said polluting substances. A further object of the sampling device of the invention is to allow a rapid and simple extraction of the adsorbing cartridge from the device.

SAMPLING DEVICE FOR VOLATILE SUBSTANCES

Field of the invention

The present invention relates to a sampling device of volatile substances dispersed in air by radial symmetry diffusion, used specifically for monitoring the pollution in working and living environments.

State of the art

Sampling of volatile substances may be currently carried out with low investment and management costs, and entirely without energy consumption, by resorting to diffusion sampling devices or samplers instead of the conventional systems based on the use of suction pumps powered by battery or by the electrical mains.

Diffusion samplers comprise axial symmetry devices and radial symmetry devices. The latter are most advantageous due to their high sampling flow rate.

A first example of a radial symmetry device is described in EP0714020.

Disadvantageously, however, such diffusion samplers can not prevent, or at least considerably reduce, the adsorption of some substances, such as for example steam, potentially interfering with the detection of the volatile substances to be analysed.

Indeed, while the atmospheric concentration of steam is normally in the order of some grams per cubic metre of air, the volatile substances which contribute to the air pollution may be present in concentrations from three to six lower orders of magnitude, thus in the order of some milligrams or micrograms per cubic metre of air.

According to the volatile substances to be determined and the adsorbing medium used in the sampler, an interference of this proportion, characterised by a concentration ratio from 1,000 to 1,000,000 times, could make retrieval and analysis of the collected substances very difficult or even make sampling impossible. This occurs, for example, if molecular sieves are used as adsorbing media, in which case the competition of steam rapidly saturates the adsorbing medium preventing the collection of the sought volatile substances.

A second example of a radial symmetry device is described in US5116577. Such device comprises an external cylindrical casing and an internal cylindrical casing which extends coaxially to the external one. The two casings define an intermediate space filled with a conversion reactant so as to convert the

substance to be monitored into a conversion product. The internal casing contains an adsorbing material suitable to collect the conversion product.

Disadvantageously, however, in order to extract the internal casing and subsequently analyse the adsorbed sample, the intermediate space must first be emptied and the remaining solid conversion reactant must be collected in a suitable container. This implies a higher complexity in extracting the internal casing and a considerable waste of time.

Furthermore, also this device cannot either prevent or at least considerably reduce the adsorption of some substances potentially interfering with the detection of the volatile substances to be analysed. An example of these interfering substances is ozone which, in high concentrations, negatively interferes on the sampling of aldehydes.

The need to make a diffusion sampling device having radial symmetry capable of overcoming the aforesaid drawbacks is therefore felt.

Summary of the invention

It is the main object of the present invention to make a sampling device for volatile polluting substances by means of radial symmetry diffusion which, in addition to allowing high sampling flow rates, allows to either prevent or considerably reduce the adsorption of some substances, such as for example steam or ozone, potentially interfering with the detection of said polluting substances.

A further object of the sampling device of the invention is to allow a rapid and simple extraction of the adsorbing cartridge from the device.

The present invention therefore proposes to obtain the aforementioned objects by making a sampling device for volatile polluting substances by radial symmetry diffusion which has the features of the claim 1.

Advantageously the device of the invention provides means for decreasing the amount of potentially interfering adsorbed substances, said means comprising an external permeation membrane of a suitable material, in a first embodiment of the device of the invention, or an adsorbing cartridge coated with a suitable reactive material in a second embodiment of said device.

In the first embodiment of the device of the invention sampling is performed in two steps: the volatile substance molecules are diffused from the air of the contaminated external environment to the internal space of the sampling device, perme-

ating through a stainless steel or other metallic material tubular membrane possibly coated with a suitable material, such as for example an hydrophobic material, and being diffused through a tubular membrane of porous material; they are finally collected by the adsorbing material arranged inside a cartridge coaxial and internal to said membranes.

In the second embodiment of the device of the invention sampling occurs by radial symmetry diffusion in two steps: the volatile substance molecules are diffused from the air of the external contaminated environment, through a first wall, into a diffusion chamber, also cylindrical; subsequently the volatile substance molecules present in the diffusion chamber are diffused towards the adsorbing material through a second wall, being the first and/or the second wall advantageously treated with a reactive material useful for the sampling procedure.

The diffusion sampler, object of the present invention, has the following advantages.

The radial symmetry of the diffusion determines, despite the small dimensions of the sampler, a high value of the parameter S/L , that is the ratio between the diffusion surface and the diffusion path length, and thus a sampling flow rate of the same order of magnitude as that obtainable in "active" sampling which provides the use of suction pumps.

The high sampling flow rate, directly proportional to the parameter S/L , compensates the opposite effect determined, in the first embodiment, by the resistance to diffusion offered by the external permeable membrane, thus allowing the determination of the atmospheric pollutants at low concentrations, e.g. 0,1 milligrams per cubic metre of air for four hour exposures.

The possibility of inserting adsorbing cartridges of different diameter inside the sampling device allows to use the techniques based on thermal desorption in addition to chemical desorption; the dimensional compatibility with the most common thermal desorbers allows to directly transfer the thermal desorption cartridges into the desorber, thus reducing handling to the minimum and consequently also the possible risks of accidental contamination of the samples.

The possibility of filling adsorbing cartridges with different materials allows to make the sampler suitable to determine various categories of volatile substances.

In particular the second embodiment of the device of the invention has the

following advantages:

- the internal porous tube can contain adsorbing materials of various nature and composition, with grain size smaller than that commonly used for volatile substance sampling, i.e. with dimensions reduced to at least 0,05 mm in diameter;
- 5 this device allows to use the aforesaid fine grain size adsorbing materials completely avoiding the drawbacks related to the high load loss, typical of "active" samplings;
- the aforesaid internal porous tube or the external porous tube, treated with a suitable reactive material, is adapted to either eliminate possible interferers or
- 10 promote chemical conversion reactions preliminary to adsorption;
- it allows a better sampling because the high thickness of the diffusion walls, constituted by porous material, significantly reduces the effect of the air speed on the diffusion sampling flow rate, which is instead relevant in other types of diffusion samplers provided with low thickness grids or membranes;
- 15 - the sampling time may vary from a few hours to a month, allowing the necessary analytic sensitivity.

The dependent claims describe preferred embodiments of the invention.

Brief description of the figures

Further features and advantages of the invention will be better apparent in the
20 light of the detailed description of preferred, but not exclusive, embodiments of a sampling device for volatile substances illustrated by way of non-limitative example, with reference to the accompanying drawings, in which:

Fig. 1 represents a longitudinal section of part of a first embodiment of the device according to the invention and lateral views of its ends;

25 Fig. 2 represents a lateral view of a component of the first embodiment of the device of the invention;

Fig. 3 represents a lateral view of a further component of the first embodiment of the device of the invention;

Fig. 4 represents a longitudinal section of the first embodiment of the complete
30 device fixed to the component of Fig. 3;

Fig. 5 represents a longitudinal section of part of a second embodiment of the device according to the invention and lateral views of its ends;

Figs. 6a and 6b respectively represent a lateral view of an alternative embodiment

of a component of the second embodiment of the device of the invention;

Fig. 7 represents a lateral view of a further component of the second embodiment of the device of the invention;

Fig. 8 represents a longitudinal section of the second embodiment of the complete device fixed to the component of Fig. 7.

Detailed description of preferred embodiments of the invention

With reference to the figures 1 to 4, a first embodiment of a radial symmetry diffusion sampling device, or simply sampler, for volatile substances is represented, indicated as a whole by reference numeral 1, comprising:

- 10 - a first tubular body or barrier 2 of permeation and/or diffusion, preferably of cylindrical shape, reusable for repeated samplings;
- a second tubular body or barrier comprising an adsorbing cartridge 3, also preferably of cylindrical shape, containing a suitable adsorbing material;
- a supporting plate 4.

15 In this description the term permeation of volatile substances means penetration and diffusion of said substances.

Tubular body 2, shown in Fig. 1, for the permeation of the volatile substances to be analysed, coming from the external environment, comprises a first porous material membrane 5 and a second permeable membrane 6, arranged coaxially and externally or internally to first membrane 5 and suitably coated with a material adapted to reduce the permeation of substances potentially interfering with the detection of the sought volatile substances.

Specifically, since the steam present in the air is an extremely interfering substance, this first preferred embodiment of the invention provides that second membrane 6 is formed by a stainless steel or other metallic material net tube, previously externally coated with a hydrophobic material, preferably a silicon resin or Teflon or, more generically, perfluoro organic polymer in shape of film.

Within second membrane 6, first membrane 5 is formed by a porous tube of porous polyethylene or porous polyolefin or any other material which gives high strength and chemical inertia.

Alternatively, second membrane 6, formed by a stainless steel or other metallic material net tube, possibly coated with a hydrophobic material, can be arranged inside the first membrane 5, formed by a porous tube of porous polyethylene or

porous polyolefin or any other material which gives high strength and chemical inertia, possibly treated with a reactive material adapted to reduce the diffusion of potentially interfering substances.

Both tube-shaped membranes 5, 6 are solidarily closed at the ends by means of a closed bottom 7 and a through head 8, preferably formed by polyethylene or other
5 polymeric material, either fixed or welded to the two tubes so as to form a single hollow cylindrical body.

Advantageously, the polyolefin tube of membrane 5 has diameter suitably designed to be inserted, in concentric and coaxial manner, inside or outside the
10 stainless steel tube of membrane 6, conferring to the whole features of strength that allow use and handling without the risk of deformation.

Adsorbing cartridge 3, positionable concentrically inside tubular body 2, contains an adsorbing material suitable for adsorbing the volatile substances which are penetrated and diffused through membranes 5, 6.

15 In a variant of this first preferred embodiment of the invention, adsorbing cartridge 3 is an electrowelded or mechanically soldered net tube of stainless steel or other metallic material, as that shown in Fig. 2, filled with an adsorbing material, powdered or liquid at high viscosity, and closed at the two ends by two cups 9, also these in stainless steel or other metallic material. The stainless steel net
20 meshes may be variable according to the grain size of the adsorbing material. In a second variant adsorbing cartridge 3 is a tube of porous polymer, preferably polyethylene.

Alternatively adsorbing cartridge 3 can be closed at one end by a stainless steel cup or a fluid-tight cap of plastic material and at the other end by a cylindrical
25 fitting-in cap, again made of plastic material.

Cartridge 3 is inserted in tubular permeation body 2 and maintained in position between a lead-in 10 arranged on the closed base or bottom 7 and the free space of through head 8 or the fitting-in cap housed in said through head. The length of the cartridge is substantially equal to that of tubular permeation body 2 but the
30 diameter is smaller with respect to said body, said cartridge being arranged in coaxial and concentric manner inside it.

Advantageously, adsorbing cartridges 3 may have suitable diameter and length to be directly inserted, without transfer of the adsorbing material, into the thermal

desorbers available on the market, provided for example with desorbing tubes of external diameter $\frac{1}{4}$ ", i.e. 0,635 mm. Thermal desorption allows to obtain higher and constant recovery efficiency and to eliminate the use of toxic reactants.

5 In the case of chemical desorption, the adsorbing material is retrieved after sampling so to analyse the substances to be determined by means of suitable chemical reactants.

In the case of chemical desorption, the adsorbing cartridge is a disposable cartridge, while it is fully reusable in thermal desorption.

10 Advantageously, the powdered adsorbing material may be of any nature and composition with a minimum grain size either equal to or lower than 0,05 mm.

Examples of adsorbing material are silica gel, activated carbon, graphitized carbons, molecular sieves.

15 With reference to the figures 5 to 8, it is represented a second embodiment of the sampling device 1', or simply sampler, for volatile substances by means of a double radial symmetry diffusion comprising:

- a first external diffusion body or barrier 2', preferably cylindrical, reusable for repeated samplings;
- a second internal diffusion body or barrier 3', also preferably cylindrical, containing a suitable adsorbing material;
- 20 - a supporting plate 4'.

The first diffusion body 2', shown in Fig. 5, for the diffusion of the volatile substances to be analysed, coming from the external environment, comprises a first tube of porous polymer, preferably polyethylene.

25 Said first tube is closed at the ends by a closed bottom 7 and by a through head 8, also these preferably made of polyethylene and fixed, for example welded, to the tube.

The second diffusion body 3' preferably comprises a second tube of porous polymer, preferably polyethylene, closed at one end by a stainless steel cup 9 (Fig. 6b) or a fluid-tight cap 15 of plastic material (Fig. 6a) and at the other end by
30 a cylindrical fitting-in cap 16, again made of plastic material.

Advantageously, the porous material tube of first body 2' has a thickness from 1 to 6 mm and the dimensions of the pores are equal to approximately 10-50 μm ; the porous material tube of second body 3' instead has a thickness of approximately

0,2-1 mm and the dimensions of the pores are equal to approximately 10-50 μm .

In a variant of this second embodiment the second body 3' is an electrowelded or mechanically soldered net tube of stainless steel or other metallic material, filled with an adsorbing material, powdered or liquid at high viscosity.

- 5 Second body 3', containing the adsorbing material, is inserted in first body 2' and held in position between the fitting-in cap 16, housed in through head 8, and the notch or lead-in 10 of the closed base or bottom 7.

Fitting-in cap 16 has shape and dimensions so as to be housed inside through head 8 of the diffusion body 2' and to further allow a convenient handling of
10 second body 3', both for the sampling and for the retrieval of adsorbing material, without there being direct contact with the same material.

Alternatively the second body 3' can be closed at the two ends by two cups in stainless steel or other metallic material. In this case the second body 3', inserted
15 ion the first body 2', is held in position between the free space of the through head 8 and the notch or lead-in 10 of the closed base or bottom 7.

The adsorbing material, powdered or liquid, is chosen so as to be suitable to adsorb the sought volatile substances which are diffused through the first and second diffusion bodies. Advantageously, the powdered adsorbing material may be of any nature and composition with a minimum grain size either equal to or
20 lower than 0,05 mm. Examples of adsorbing material are silica gel, magnesium silicate, porous polymers, molecular sieves, quartz, activated carbon, graphitized carbons.

Preferably the second body 3' has a length substantially equal to that of first body 2' and is arranged in coaxial and concentric manner with respect to the latter.

- 25 Advantageously, either first diffusion body or barrier 2', or second diffusion body or barrier 3', or both said bodies are appropriately treated with a reactive material adapted to eliminate or decrease the amount of possible substances potentially interfering with the detection of the sought volatile substances and/or to promote chemical conversion reactions preliminary to the adsorption of said volatile
30 substances.

Specifically, since the ozone present in air is an extremely interfering substance, which depresses the sampling of formaldehyde and of other aldehydes, this second preferred embodiment of the invention provides in a first variant that

second body 3' is formed by a porous polymeric material tube preventively treated with an ozone reducing material, preferably potassium iodide.

Furthermore, since nitrogen monoxide as such is not efficiently adsorbed by the currently available adsorbing materials, while it may be adsorbed after oxidation to nitrogen dioxide, a further variant of the second preferred embodiment of the invention provides that second body 2' is formed by a porous polymeric material tube preventively treated with an oxidising material, preferably potassium permanganate.

An advantageous alternative embodiment of the invention provides that first body 2' comprises a first reactive material adapted to eliminate possible substances potentially interfering with the detection of the sought volatile substances, and that second tubular body 3' comprises a second reactive material adapted to promote chemical conversion reactions preliminary to the adsorption of said volatile substances, or vice versa.

The reactive material is advantageous arranged in form of crystals inside the pores and on the external surface of first body 2' and/or of second body 3', by means of a specific deposition procedure.

The adsorbing material is retrieved after sampling so to analyse the substances to be determined by means of suitable chemical reactants.

The "equivalent pumping" capacity of the sampler of the invention is approximately 30-100 cm³/min of air, its total weight is equal to approximately 20-30 g, and its limit of analytic sensitivity is in the order of 1 part per billion (ppb) for a seven-day exposure.

The constructive material of the sampler, including diffusive surfaces, is preferably high-thickness polyethylene or any other material capable of conferring high strength and chemical inertia.

For both preferred embodiments the assembly of the sampler of the invention is completed by fixing the first body 2, 2', containing the second body or adsorbing cartridge 3, 3', to the supporting plate 4, 4', preferably produced in polycarbonate or polyethylene, in the manner shown for example in Fig. 4 and 8. In this case, through head 8 is provided with an external threading 12 for fixing the sampler to the corresponding threaded seat 13 provided on supporting plate 4, 4'. In an alternative embodiment, the fixing between sampler of the invention and plate

may simply be of fitting-in type with plate 4, 4' acting as cap.

Supporting plate 4, 4' is preferably but not necessarily triangular in shape, as shown in Fig. 3 or 7. Such plate 4, 4', provided with one or more housings for clips 11, preferably three in number, allows to apply the sampler to people's clothes or to fix it to a suitable support provided in the various environments.

5

CLAIMS

1. A sampling device for volatile polluting substances by radial symmetry diffusion comprising
 - at least one first hollow diffusion or permeation body (2, 2') of said volatile
 - 5 substances from an external environment to inside said first body,
 - a second hollow diffusion body (3, 3'), positionable inside said at least one first body (2, 2'), containing an adsorbing material adapted to adsorb said volatile substances,
 - characterised in that at least one of said first body (2, 2') or second body (3, 3')
 - 10 comprises means for decreasing the amount of possible substances potentially interfering with the detection of the volatile polluting substances.
2. A device according to claim 1, wherein said first body (2) comprises a first membrane (5) of porous material and said means for decreasing the amount of possible potentially interfering substances comprise a second permeable
- 15 membrane (6), arranged coaxially and externally or internally to the first membrane (5) and coated with a predetermined material.
3. A device according to claim 2, wherein said predetermined material is a hydrophobic material.
4. A device according to claim 3, wherein said hydrophobic material is silicon resin
- 20 in shape of film or teflon or perfluoro organic polymer in shape of film.
5. A device according to any of the preceding claims, wherein the second membrane (6) is formed by a metallic material net tube.
6. A device according to claim 5, wherein the first membrane (5) is formed by a porous tube in porous polyethylene or polyolefin.
- 25 7. A device according to claim 6, wherein said first and second membrane (5, 6) are solidarily closed at the ends by means of a closed bottom (7) and a through head (8).
8. A device according to any of the preceding claims, wherein said second hollow body comprises an adsorbing cartridge (3) formed by an electrowelded or
- 30 mechanically soldered metallic material net tube, or formed by a tube of porous polymer.
9. A device according to claim 8, wherein the adsorbing cartridge (3) is suitably sized to be directly inserted, without transfer of the adsorbing material, in thermal

desorbers.

10. A device according to claim 1, wherein said means for decreasing the amount of possible potentially interfering substances comprise a predetermined reactive material suitable to possibly promote chemical conversion reactions preliminary to the adsorption of said volatile substances.

11. A device according to claim 10, wherein said predetermined reactive material is a reducing material, preferably potassium iodide.

12. A device according to claim 10, wherein said predetermined reactive material is an oxidising material, preferably potassium permanganate.

13. A device according to claim 11 or 12, wherein said first and second bodies (2', 3') comprise respectively a porous polymer tube.

14. A device according to claim 13, wherein said porous polymer is polyethylene.

15. A device according to claim 13 or 14, wherein the reactive material is arranged in form of crystals inside the pores and on the external surface of the porous polymer of the first body (2') and/or of the second body (3').

16. A device according to any of the preceding claims from 10 to 15, wherein said first body (2') is closed at the ends by means of a closed bottom (7) and a through head (8).

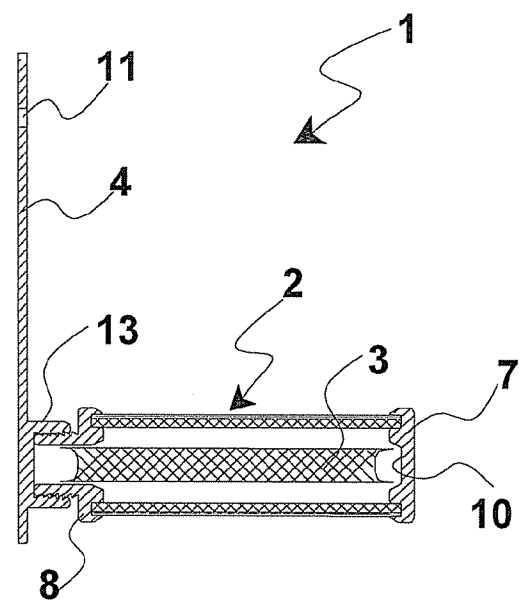
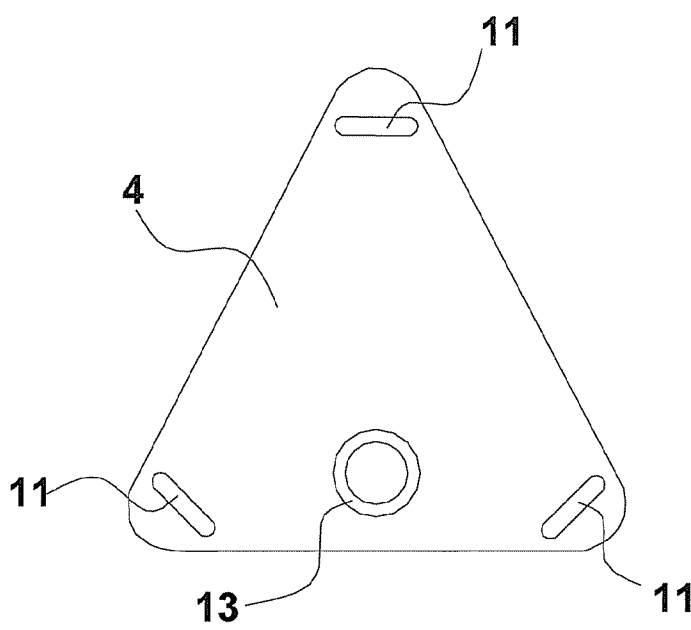
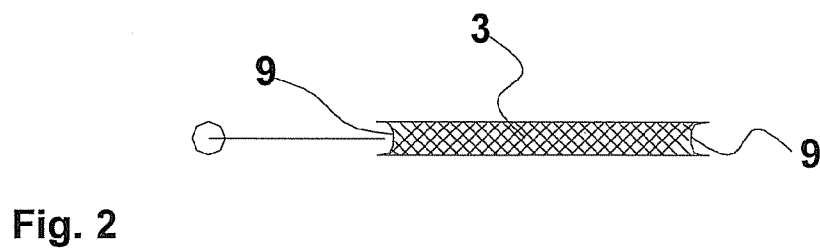
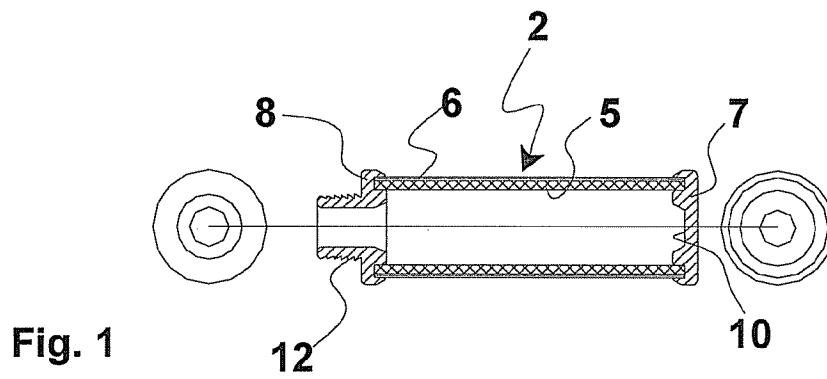
17. A device according to any of the preceding claims from 10 to 15, wherein the adsorbing material may be either powdered, with minimum grain size equal to or smaller than 0,05 mm, or liquid.

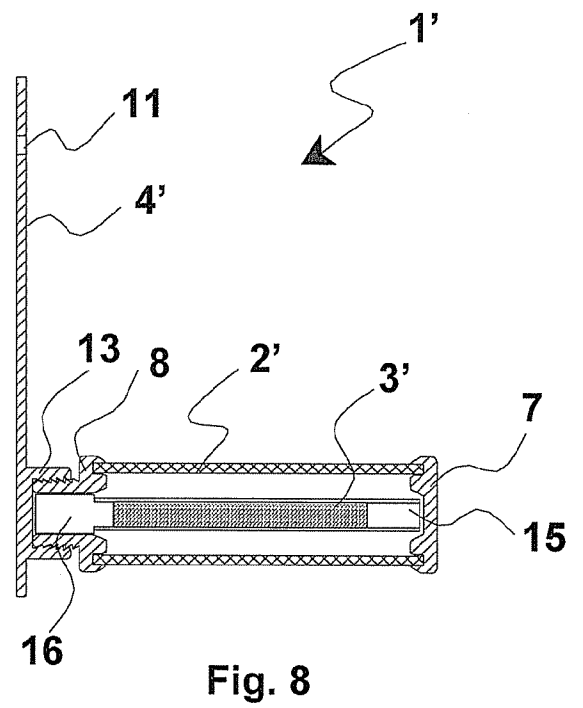
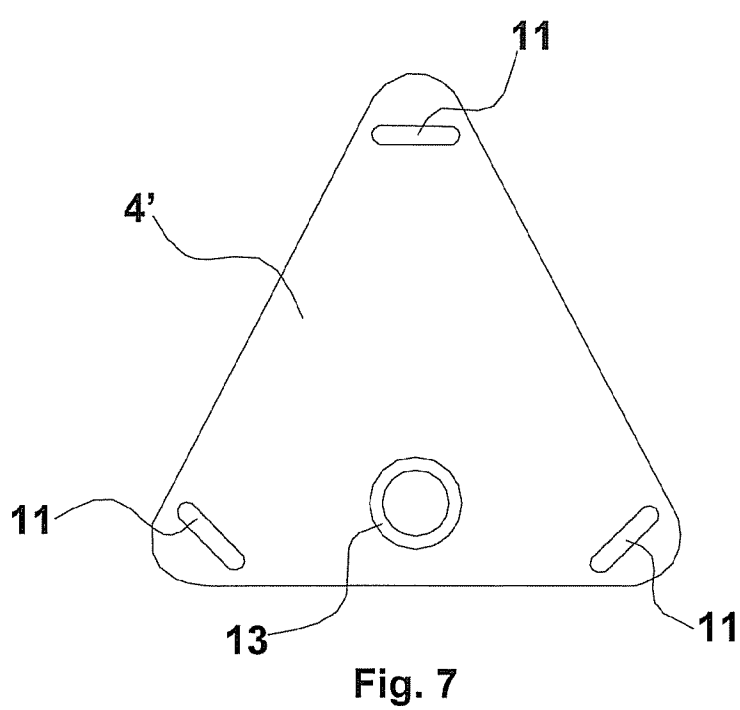
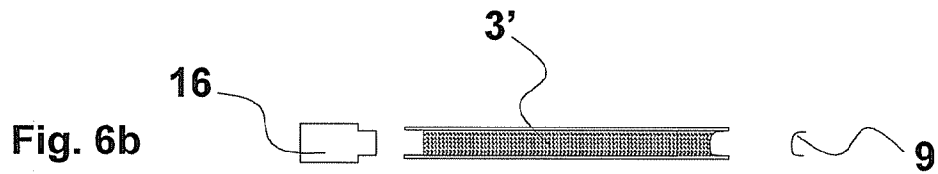
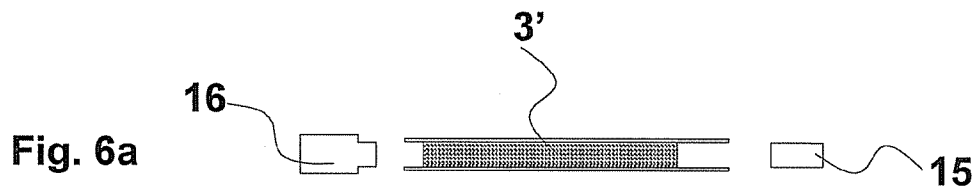
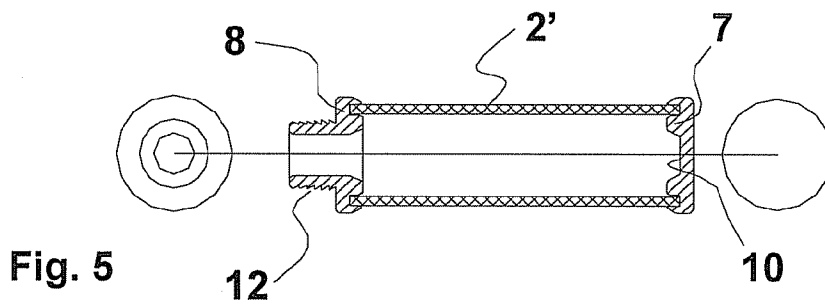
18. A device according to claim 7 or 16, wherein said second body (3, 3') is closed at the two ends by two metallic material cups (9), or is closed at a first end by a fitting-in cap (16), adapted to be housed in the through head (8) and to allow the handling of the second body without any direct contact with the same, and at a second end by a metallic material cup (9) or by a cylindrical cap (15).

19. A device according to claim 18, wherein the second body (3, 3') is adapted to be inserted in the first body (2, 2') through the through head (8) and held in position between a lead-in (10), provided in the closed bottom (7), and the fitting-in cap (16) or the free space of the through head (8).

20. A device according to any of the preceding claims, wherein there is provided a supporting plate (4, 4') adapted for a fixing of the device and provided with one or more housings (11) for clips so as to apply said device to people's clothes or to a

suitable support provided in the various environments.





INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2007/053841

A. CLASSIFICATION OF SUBJECT MATTER

INV. G01N1/22

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)

G01N

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)

EPO-Internal, WPI Data, INSPEC, COMPENDEX, BIOSIS, EMBASE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 116 577 A (EICKELER EDGAR [DE] ET AL) 26 May 1992 (1992-05-26) cited in the application	1,10,12
Y	the whole document	1-20
Y	----- EP 0 714 020 A2 (FONDAZIONE SALVATORE MAUGERI [IT] FONDAZIONE SALVATORE MAUGERI) 29 May 1996 (1996-05-29) the whole document	1-20
A	----- US 2003/215958 A1 (KIN PONG BOON [SG] ET AL) 20 November 2003 (2003-11-20) figure 1	1-20



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

31 July 2007

Date of mailing of the international search report

10/08/2007

Name and mailing address of the ISA/

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Timonen, Tuomo

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2007/053841

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