

ePCT - Cover Letter

ePCT Action: Submit Chapter II Demand

Competent IPEA:

BR

IA Number:

PCT/BR2024/050275

The following documents were submitted as attachments:

- Chapter II Demand : 1 document
- Fee-related Correspondence: 1 document
- Article 34 Amendments: 2 documents
- Letter accompanying amendments under Article 34: 1 document

Signature:	/FERNANDA L. EMERENCIANO/
Signatory:	VSOL ANÁLISES AMBIENTAIS LTDA. (Applicant)
Name of signatory:	FERNANDA LINS EMERENCIANO
Position in company:	LEGAL REPRESENTATIVE
Date:	13 December 2024 14:22:31 BRT
User Name:	OFFICE ICAMP
Authentication:	Sign-in with strong authentication

PCT

DEMAND

under Article 31 of the Patent Cooperation Treaty:
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty.

For International Preliminary Examining Authority use only

Identification of IPEA: BR	Date of receipt of DEMAND: 13 December 2024(13.12.2024)
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Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION		Applicant's or agent's file reference VSOL01
International application No. PCT/BR2024/050275	International filing date 26 June 2024(26.06.2024)	(Earliest) Priority date 04 September 2023(04.09.2023)
Title of invention TUBE FOR VOLATILE AND SEMI-VOLATILE COMPOUND SAMPLING AND SAMPLING PROCESS		
Box No. II APPLICANT(S)		
Name and address: VSOL ANÁLISES AMBIENTAIS LTDA. RUA AGOSTINO TOGNERI, Nº 115, CONJ. A, BAIRRO JURUBATUBA 04.690-090 SÃO PAULO (SP) Brazil		Telephone No. +55 (19) 3255-7899
		Facsimile No.
		Applicant's registration No. with the Office 04.046.780/0001-61
Nationality: Brazil	Residence: Brazil	
E-mail authorization: Marking one of the check-boxes below authorizes the International Bureau and the International Preliminary Examining Authority to use the e-mail address indicated in this Box to send notifications issued in respect of this international application if those offices are willing to do so. ☐ as advance copies followed by paper notifications; or ☒ exclusively in electronic form (no paper notifications will be sent). E-mail address: brazil@icamp.com.br		
☐ Further applicants are indicated on a continuation sheet.		

Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The following person is ☒ agent ☐ common representative

and ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.

☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.

☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.

Name and address:

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54.675.251/0001-03

E-mail authorization: Marking one of the check-boxes below authorizes the International Bureau and the International Preliminary Examining Authority to use the e-mail address indicated in this Box to send notifications issued in respect of this international application if those offices are willing to do so.

- ☐ as advance copies followed by paper notifications; or
- ☒ exclusively in electronic form (no paper notifications will be sent).

E-mail address: icamp@icamp.com.br

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION**Statement concerning amendments:***

1. The applicant wishes the international preliminary examination to start on the basis of:

- the description ☐ as originally filed, or
☒ as amended under Article 34
- the sequence listing (if any) ☐ as originally filed, or
☐ as amended under Article 34
- the claims ☐ as originally filed, or
☐ as amended under Article 19, and/or
☒ as amended under Article 34
- the drawings (if any) ☒ as originally filed, or
☐ as amended under Article 34

2. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.
3. ☐ Where the IPEA wishes to start the international preliminary examination at the same time as the international search in accordance with Rule 69.1(b), the applicant requests the IPEA **to postpone** the start of the international preliminary examination until the expiration of the applicable time limit under Rule 69.1(d).
4. ☐ The applicant expressly requests **to postpone** the start of the international preliminary examination until the expiration of the applicable time limit under Rule 54bis.1(a).

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: English

- ☒ which is the language in which the international application was filed.
- ☐ which is the language of a translation furnished for the purposes of international search.
- ☒ which is the language of publication of the international application.
- ☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

Box No. V ELECTION OF STATES

The filing of this demand constitutes the election of all Contracting States which are designated and are bound by Chapter II of the PCT.

Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

- Fee-related Correspondence: 1 document
- Article 34 Amendments: 2 documents
- Letter accompanying amendments under Article 34: 1 document

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).

/FERNANDA L. EMERENCIANO/

Name: **VSOL ANÁLISES AMBIENTAIS LTDA.**

Name of Signatory: **FERNANDA LINS EMERENCIANO**

Capacity: **LEGAL REPRESENTATIVE**

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND: 13 December 2024(13.12.2024)	
2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):	
3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply. ☐ The applicant has been informed accordingly.	6. ☐ The date of receipt of the demand is AFTER the expiration of the time limit under Rule 54bis.1(a) and item 7 or 8, below, does not apply.
4. ☐ The date of receipt of the demand is WITHIN the time limit of 19 months from the priority date as extended by virtue of Rule 80.5.	7. ☐ The date of receipt of the demand is WITHIN the time limit under Rule 54bis.1(a) as extended by virtue of Rule 80.5.
5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rules 82 or 82quater.	8. ☐ Although the date of receipt of the demand is after the expiration of the time limit under Rule 54bis.1(a), the delay in arrival is EXCUSED pursuant to Rules 82 or 82quater.

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Demand received from IPEA on:

PCT

FEE CALCULATION SHEET**Annex to the Demand**

International application No. PCT/BR2024/050275	For International Preliminary Examining Authority use only Date stamp of the IPEA	
Applicant's or agent's file reference VSOL01		
Applicant VSOL ANÁLISES AMBIENTAIS LTDA.		
CALCULATION OF PRESCRIBED FEES		
1. Preliminary examination fee	630.0 (BRL) P	
2. Handling fee (Applicants from certain States are entitled to a reduction of 90% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 10% of the handling fee.)	0 () H	
3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box		
		TOTAL
MODE OF PAYMENT (Not all modes of payment may be available at all IPEAs)		
☐ authorization to charge deposit or current account with the IPEA (see below) ☐ credit card (details should be furnished separately and not included on this sheet)		
☐ cheque ☐ revenue stamps		
☐ postal money order ☐ cash		
☐ bank transfer ☐ other(specify):		
AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT OR CURRENT ACCOUNT (This mode of payment may not be available at all IPEAs)		
☐ Authorization to charge the total fees indicated above. IPEA/		
☐ (This check-box may be marked only if the conditions for deposit or current accounts of the IPEA so permit) Deposit or Current Account No.:		
Authorization to charge any deficiency or credit any overpayment in the total fees indicated above. Date:		
Name:		
Signature:		

RESPONSE TO PCT WRITTEN OPINION

INTERNATIOANL APPPLICATION: **PCT/BR2024/050275**

APPLICANT: **VSOL ANÁLISES AMBIENTAIS LTDA.**

In response to the Written Opinion issued by the Patent Cooperation Treaty (PCT) authority,
the Applicant submits the following analyses and amendments:

WRITTEN RESPONSE

The examining authority states that:

1. **Claim 1:** While new, Claim 1 lacks an inventive step and therefore does not comply with PCT Article 33(3).
2. **Claims 2 and 3:** Claims 2 and 3 are also new but lack an inventive step in view of the cited references. Consequently, they do not comply with PCT Article 33(3). Additionally, the claims do not comply with PCT Article 6 because the relationship between Claims 2 and 3 and Claim 1 is not explicitly stated in the claims preamble.

The Applicant respectfully disagrees with the Examining Authority's opinion and provides arguments below to demonstrate the inventive step of the invention.

Amendments to the specification and claims are also provided to facilitate the grant of the application. The references cited are as below:

- D1: BR 20 2019 014185
- D2: CN 103995069

Document **D1** reveals that

Document D1 refers to passive sampler of soil vapors with semi-permeable membrane in PTFE (polytetrafluoroethylene) (3) and amber bottle traceable. The present utility model patent deals with a passive sampler for investigation and measurement of volatile organic compounds (COVs) and semi-volatile (COSVs) in contaminated areas, composed of an amber bottle with a traceable code, of varying dimensions, with a lid consisting of a semipermeable membrane of PTFE and with specific formulation of polymeric resins and molecular sieves. The formulation of the sorbent materials adsorbs the organic elements present in the soil and the semipermeable membrane avoids interference by the presence of water. the amber bottle (7), with traceable code, considerably reduces the degradation of these compounds by the presence of light. The passive sampler comprises an amber bottle (7) with a traceable serial number (8) with collection cap (2). A hollow center (1) is connected to semi-permeable membrane (3) in PTFE with sealing washer (4). The mesh supports are provided with adsorbent material (5a to 5f).

While Document **D2** states that

Document D2 refers to micro-needle capture device and using method of the invention relates to a volatile semi volatile organic emergency monitor, which is connected with the airtight syringe through luer connector by micro needle. The device has a collecting micro-needle (6) connected with an air injector (7) through a luer connector (8). The collecting micro-needle is provided with a needle point (1), a needle (2) and a needle base (5), where outer diameter of the needle is 0.72 mm and inner diameter of the needle is 0.4 mm. The needle is equipped with a spring plug (3) of an adsorbent bed layer (4), where the adsorbent bed layer is an active carbon fiber layer and length of the adsorbent bed layer is 10-30 mm.

As stated in the abstract of document **D1**, the sampler object of this document is a passive sampler. Therefore, to perform active sampling, an adequate amount and composition of sorbents are required.

Although some analogies can be found between **D1** and the application, the teachings of **D1** do not lead to the described invention since they do not suggest or describe a device capable of performing the collection of samples for both active and passive environmental sampling.

The device of the invention is the only disclosed instrument that can be easily adapted to take environmental samples actively or passively.

The sample tube (1) of the invention has two caps (5), one distal and one proximal to the direction of flow (3). This allows the tube to be adapted to:

- a) active sampling through removal of cap (5) at the end of tube (1) coincident with the direction indicated by flow arrow (3) and then attachment of tube (1) to syringe (8), removal of the other cap (5) for attachment to sampling point and sample catchment. During catchment, check valve (9) only allows flow in the direction for pulling and pushing the syringe plunger;
- b) passive sampling, by removing cap (5) from tube (1) section opposite to the flow direction indicated by flow arrow (3), connecting tube (1) to sampling cap set (10) and positioning the sample tube at the location to be sampled. After the sampling period, tube (1) is removed and cap (5) is replaced. In this mode only one cap (5) is removed.

The concept underlying tube (1) of the invention is a tube filled with hydrophobic and oleophobic adsorbents and sealed with two caps (5), one at each end.

By selectively removing one or both caps (5), two different samplers can be easily assembled, offering significant practicality and cost savings. This is particularly valuable in countries with limited research resources.

This practical and economic advantage is neither suggested nor described in **D1** or **D2**.

D1 is limited to passive sampling, which restricts the types of samples that can be collected.

Regarding document **D2**, it describes a complex device with a microneedle connected to an air injector, designed for use as an emergency monitor. This patent document differs from the present application in its purpose and is not relevant to the invention.

Document D2 discloses a different approach that is not relevant to the invention. Its teachings would not guide a skilled person towards the claimed solution, but rather away from it.

Furthermore, neither D1 nor D2 can be adapted to perform both passive and active sampling through simple modifications, as is possible with sampling tube (1) of the present invention. For example, the syringe and microneedle of D2 cannot be rearranged to collect soil samples because soil VOCs (volatile organic compounds) and SVOCs (semi-volatile organic compounds) require different adsorbents than those used for air VOCs and SVOCs.

On the other hand, the Applicant is aware of some inconsistencies and lack of clarity in the specification and claims. Therefore, specification and claims have been amended to comply with PCT Article 33(3) and are attached hereto in MARKED-UP and CLEAN files.

The amendments are based on good patent practices, such as consistently using the same word to indicate the same element of the device. For example, the word "tube" was initially translated as both "tube" and "pipe." Although synonyms, only "tube" has been retained in the specification and claims to refer to sample tube (1) for clarity.

Also, the English syntax has been improved for clarity.

The few amendments made to the specification and/or claims that might appear to be new matter are based on information provided in the figures.

Thus, for example:

- I. In paragraph [017] the expression “as below” has been added for the sake of clarity, to designate that the following paragraphs would describe the process steps used in active sampling.
- II. Item I. of the same paragraph was rewritten for the sake of English syntax and of course clarity, since poor syntax impairs clarity.
- III. In paragraph [026], an unrelated phrase relative to adsorbent particle size has been deleted.
- IV. In line 4 of paragraph [021] of the as-filed Application the phrase “membrane (13) sandwiched between silicone tube (11) and rigid support (12), said sampling cap set (10) ...” has been added to better define the sampling cap set (10) used in passive sampling in terms of the position of each of the elements (11), (12), (13).
- V. The phrase is based on Figures 9, 10 and 11 where it can be neatly seen the location of membrane (13) in between silicone tube (11) and rigid support (12).

The Applicant asserts that Figures 9, 10, and 11 provide sufficient support for the added phrase in paragraph [021], and its inclusion does not constitute new matter.

The Applicant believes further that this reading brings more clarity to the invention since it better defines the relative positions of elements (11), (12) and (13) of sampling cap set (10) used in passive sampling with the aid of the inventive device.

The same amendment has been used in new claim 2.

The Applicant believes that the relative positions of elements (11), (12) and (13), being now explicit in the specification make sufficient antecedent basis for new claim 2.

Claim 1 has been amended to place before the characterizing expression the feature of presence of adsorbents in the interior of the tube, which is common to all state-of-the-art samplers.

Also, claim 1 now expresses the two possibilities of sampling provided by the inventive device: active and passive sampling.

To comply with PCT Article 6, Claims 3 and 4, process claims, have been duly connected to the device claimed in claim 1.

For the sake of uniformity of notation, former process steps A. to E. of claim 4 have been renumbered as i. to v. to be in agreement with notation of corresponding process steps of claim 3.

New claim 2, dependent from claim 1, has been added to explicit the constituting elements of sampling cap set (10).

Although sampling cap set (10) is similar to other sampling caps for passive sampling, it is believed that the fact that it is attached to the tube (1) – caps (5) set is sufficient to provide an inventive step to the inventive device.

Consider also that environmental sampling equipment are severely limited in design, since in active sampling, samples should be taken by direct aspiration, so a syringe or similar device is practically mandatory.

On the other hand, in passive sampling the equipment should be designed to take gases generally from soil, so a tube and a membrane are also highly recommended.

In a sector with such few possibilities of creative solutions, the dual sampling tube of the invention brings a breadth of fresh air in that it is able to perform both active and passive sampling through an ingenious solution, that is:

- a) by removal of both caps (5) from tube (1) ends, coupling of syringe (8) to one end of tube (1) and check valve system (9) to the other end of tube (1) in case of active sampling;

- b) by removal of cap (5) end opposite to the direction of flow arrow (3), coupling of sampling cap set (10) to tube (1) in case of passive sampling.

Last but not least, flow arrow (3) has a crucial role in the equipment working since it indicates not only which of the two caps (5) (or both) should be removed to install one or other version of the equipment, but in active sampling, where both caps (5) are removed to assemble the device, which cap remove first. The order of removal is dictated by the relative position of each cap to flow arrow (3).

So: in active sampling: at first remove cap (5) in the direction coincident with flow arrow (3), then the other cap (5);

In passive sampling, only one cap (5) is removed, and it is cap (5) opposite to the direction of flow arrow (3).

The Applicant believes the inventive subtleties and practical advantages of this two-in-one device clearly distinguish it from the prior art. The amended specification and claims further support the grant of this application.

Encl.: Specification and claims MARKED-UP

Specification and claims CLEAN

TUBE FOR VOLATILE AND SEMI-VOLATILE COMPOUNDS SAMPLING AND SAMPLING PROCESS

[001] This invention is located in the field of environmental chemistry, specifically instruments for sampling and analyzing air in adsorbent materials to determine the chemical composition of collected samples, specifically a tube with a hydrophobic adsorbent element in a hybrid active and passive category for sampling volatile and semi-volatile compounds in environmental contamination investigations or for prospecting natural resource sources such as oil in its various forms and sampling processes.

[002] The management of contaminated areas is a sector that is more than 30 years old and during its evolution several tools were created to assist in the investigations necessary for management. Despite this, there is still a great shortage of economically viable and technically appropriate tools for environmental contamination assessments in contaminant scan mode. Tools for environmental investigation in scan mode require versatility of application and list of compounds, performance in different environmental conditions, such as very humid environments or even the presence of water that can affect the collection process and a reduced cost when compared to other techniques.

[003] An in-depth analysis of the existing technology by this inventor reveals a lack of volatile and semi-volatile compound samplers for hybrid active or passive sampling with quantitative analysis and solvent sample extraction, such as the Waterloo Membrane Sampler (WMS), which is a sampling device, a glassy container containing an adsorbent element with a lid and a vapor/gas permeable membrane, for quantitative assessment of volatile organic compounds by passive sampling only. The analysis is performed by a solvent sample preparation or by thermal desorption, processes which are followed by a gas chromatography analysis.

[004] Also, the US EPA TO-17 methodology discloses an analytical and sampling method disclosed by ~~the~~ US EPA for volatile organic compound analyses for quantitative evaluation by active-only sampling. Sampling is performed in tubes containing one or more adsorbent elements. The extraction is performed only by ~~the~~ thermal desorption methodology ~~of the tube~~ and then by gas chromatography analysis.

[005] Furthermore, the USEPA TO-3C methodology is an analytical and sampling method disclosed by ~~the~~ US EPA for analysis of semi-volatile organic compounds for quantitative evaluation by active-only sampling. Sampling is performed in tubes containing adsorbent elements and also with a polyurethane filter. Preparation is done by extraction using solvents and the Soxhlet process. The analysis is performed by gas chromatography.

[006] Finally, the NIOSH methodology is an analytical method for occupational and sampling assessments described by NIOSH for analyses of a limited list of volatile organic compounds. Sampling is active and glass tubes containing coconut activated carbon are used. There is a major limitation in this case as coconut activated carbon is not hydrophobic and can be interfered with by moisture when applied to soil air sampling, typically with high moisture contents.

[007] In view of the gaps in the technique, the proposed invention is a sampling system ~~equipped with~~ comprising a tube (1) with a mixture of passive or active hybrid category adsorbent materials for sampling volatile and semi-volatile compounds for investigations of environmental contamination or for prospecting natural resource sources such as oil in its various forms and its sampling process.

[008] The invention is a tube (1) composed of metallic, glassy or polymeric material, rigid and inert to the substances or compounds to be sampled and analyzed. The outer surface of ~~the pipe~~ the tube (1) has engravings of an arrow (3) indicating the sampling flow and ~~the pipe~~ the tube (1) serial number (4) for traceability.

[009] ~~The pipe~~ Tube (1) has two polyethylene caps (5) for closure.

[010] Internally it is composed of a mixture of hydrophobic activated carbon spheres (2) of different sizes, from 0.4 to 0.8mm and surface area from 1000 to 1300m²/g that are fixed with the ~~use~~ aid of metallic or vitreous fabrics or other inert materials (6) to the substances or compounds that will be sampled and analyzed. The set also has two stainless steel ~~retainer~~ retaining springs (7) that prevent the movement of the internal elements inside ~~the said~~ tube during active sampling.

[011] The invention may be better understood with the aid of the attached ~~f~~Figures.

[012] Figure 1 shows a front view of ~~the sampler~~ sample tube (1).

[013] Figure 2 reveals a side view of ~~the sampler~~ sample tube (1) without a cap.

[014] Figure 3 shows the internal composition of ~~the~~ tube (1).

[015] Figure 4 shows an expanded view of ~~the~~ tube (1) and internal composition. For active sampling, in addition to ~~the sampler~~ sample tube (1), a syringe (8) coupled to a check valve system (9) is used.

[016] Figure 5 shows ~~the~~ syringe (8) and ~~the~~ check valve system (9).

[017] ~~The system facilitates the~~ Sampling process is facilitated by avoiding errors during the process, as there is no need to disconnect the sampler sample tube (1) from the system. For active sampling, ~~the sampling~~ sample tube (1), ~~the~~ syringe (8) and ~~the~~ check valve system (9) are used, as below:

- i. Remove ~~the~~ cap (5) of the from sampler sample tube ~~part-(1)~~ section ~~to where the~~ coincident with the direction of flow arrow (3) ~~points~~;
- ii. Connect ~~the sampler~~ sample tube (1) to ~~the~~ syringe (8) as shown in Figure 6;
- iii. Remove ~~the~~ cap (5) still remaining from ~~the sampler~~ sample tube (1) and connect to the sampling point;

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- iv. Pull and push ~~the~~ syringe (8) plunger according to the required volume to be sampled. The check valve system (9) only allows airflow in the flows indicated by ~~figures~~ Figure 7, pulling the plunger, and Figure 8, pushing the plunger;
- v. Disconnect ~~sampler~~ sample tube (1);
- vi. Replace ~~the~~ both caps (5).

[018] Figure 6 shows ~~the~~ tube (1) connected to ~~the~~ syringe (8) and valve system (9).

[019] Figure 7 shows the flow by pulling ~~the~~ syringe (9) plunger when air from the sampled point passes through ~~the~~ sample tube (1).

[020] Figure 8 shows the flow pushing ~~the~~ syringe (9) plunger when air is relieved by ~~the~~ side check valve (9).

[021] For passive sampling only a specific sampling cap set (10) is used. It is a cap ~~that has~~ with a silicone tube (11) section, ~~and~~ a rigid metallic or polymeric support (12) and a hydrophobic and ~~olephobic~~ oleophobic membrane (13) sandwiched between silicone tube (11) and rigid support (12), said sampling cap set (10) being composed made of polytetrafluoroethylene (PTFE) or acrylic copolymers-copolymers, which allows the free passage of the compounds and/or substances of interest and ~~which~~ prevents the formation of barriers by the presence of water in its liquid state in the catchment region of the compounds and/or substances of interest. In addition, membranes drastically reduce the passage of water in vapor form, which can cause analytical problems such as low sensitivity.

[022] Figure 9 illustrates ~~the~~ sampling cap set (10) used for the passive sampling process.

[023] For passive sampling, ~~the sampler~~ sample tube (1) and ~~the~~ sampling cap (10) are used, as below:

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- A. Remove ~~the~~ cap (5) from the ~~counter sampler~~ sampler sample tube ~~part~~ (1) section opposite to the flow direction indicated by ~~where the flow arrow~~ (3);
- B. Connect ~~the sampler~~ sample tube (1) to ~~the~~ sampling cap set (10);
Position ~~the sampler~~ sample tube (1) at the location to be sampled. For example: Positioned within a building for indoor air assessment or ground burial for soil air assessment;
- C. After the sampling period, normally 7-14 days, remove ~~sampler~~ sample tube (1); and
- D. Replace cap (5)

[024] Figure 10 shows the internal composition of ~~the pipe~~ sample tube (1) assembled for passive sampling.

[025] Figure 11 demonstrates a front view of ~~the sampler~~ sample tube (1) mounted for passive sampling.

[026] In summary, the new system-combined with the corresponding process is the only sampler designed for active or passive sampling. For this type of sampler, it is possible to analyze volatile compounds both-by thermal desorption and solvent extraction. Furthermore, ~~the~~ extraction can be performed using different solvents and, therefore, allows the inclusion of analysis of semi-volatile compounds as well as multiple analyses including dilutions that are not suitable for the thermal desorption method and the adsorbent elements ~~composed of a mixture of hydrophobic activated carbon spheres (2) of different sizes, from 0.4 to 0.8mm and surface area from 1000 to 1300m²/g and~~ selected for the sampling of various organic compounds. The hydrophobicity of the adsorbent elements inhibits the interference of moisture by saturation and therefore does not generate false negatives.

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[027] This invention is not limited to the representations commented or illustrated herein, and should be understood in its broad scope. Many modifications and other representations of the invention will come to the mind of one skilled in the art to which this invention belongs, having the benefit of the teaching presented in the previous descriptions and attached drawings. Furthermore, it is to be understood that the invention is not limited to the specific form disclosed, and that modifications and other forms are understood to be included within the scope of the appended claims.

Although specific terms are employed herein, they are used only in a generic and descriptive manner and not for the purpose of limitation

CLAIMS

1. ~~(currently amended)~~ ~~SAMPLE TUBE~~ Sample tube (1) containing adsorbent elements for volatile and semi-volatile compounds sampling, provided with serial number (4) and containing an adsorbent (2), characterized by its components comprising: a rigid, inert tube (1) of metallic, glassy or polymeric material, rigid and inert, in which its external the outer surface of same has engravings of being engraved with an arrow (3); and tube serial number (4); two caps, preferably in polyethylene (5), with a hydrophobic and oleophobic membrane (13), composed of polytetrafluoroethylene (PTFE) or acrylic copolymers; and internally, it is composed of as adsorbent, a mixture of hydrophobic activated carbon spheres (2) of different sizes, from 0.4 to 0.8 mm and surface area from 1000 to 1300m²/g, fixed in metallic and vitreous fabrics (6); and two ~~retainer~~ retaining springs (7), further comprising,
 - a) for active sampling, a syringe (8) and a check valve (9) to be attached to sample tube (1) after removal of cap (5) from sample tube (1) section in the direction indicated by flow arrow (3); and
 - b) for passive sampling, sampling cap set (10) comprising a silicone tube (11) section, a rigid support (12) and a hydrophobic and oleophobic membrane (13), to be attached to sample tube (1) after removal of cap (5) from sample tube (1) section opposite to the flow direction indicated by flow arrow (3).
2. ~~(new)~~ Sample tube (1) according to claim 1b), characterized in that sampling cap set (10) comprises a silicone tube (11) section, a rigid support (12) and a hydrophobic, oleophobic membrane (13) sandwiched between silicone tube (11) and rigid support (12).
3. ~~(currently amended)~~ ~~PROCESS~~ A process for active sampling of volatile and semi-volatile compounds to be carried out with the aid of sample tube according to claim 1, characterized by its comprising the following steps:

- i. ~~Removing the cap (5) of the from sampler sample tube part (1) section to~~
where the coincident with the direction indicated by flow arrow (3) points;
 - ii. ~~Connecting the sampler sample tube (1) to the syringe (8); as shown in~~
Figure 6;
 - iii. ~~Removing the cap (5) still remaining from the sampler sample tube (1)~~
and connecting same to the sampling point;
 - iv. ~~Pulling and pushing the syringe (8) plunger according to the required~~
volume to be sampled, with The check valve system (9) only allows
allowing airflow in the flows indicated by figures Figure 7, direction for
pulling the plunger, and Figure 8, for and pushing the plunger;
 - v. ~~Disconnecting sampler sample tube (1); and~~
 - vi. Replacing both caps (5).
4. ~~(currently amended) PROCESS A process for passive sampling of volatile and~~
semi-volatile compounds to be carried out with the aid of sample tube according to
claim 1, characterized by its its steps: comprising the following steps:
- ~~A. i. Remove~~ Removing the cap (5) from the counter sampler sample tube
part (1) section opposite to the flow direction indicated by where the flow
arrow (3);
 - ~~B. ii. Connecting the sampler sample tube (1) to the sampling cap set (10);~~
 - ~~C. iii. Positioning the sampler sample tube (1) at the location to be sampled;~~
~~For example: Positioned within a building for indoor air assessment or~~
~~ground burial for soil air assessment.~~
 - ~~D. iv. After the sampling period, normally of 7-14 days, removing sampler~~
sample tube (1); and
 - ~~E. v. Replace~~ Replacing cap (5).

TUBE FOR VOLATILE AND SEMI-VOLATILE COMPOUNDS SAMPLING AND SAMPLING PROCESS

[001] This invention is located in the field of environmental chemistry, specifically instruments for sampling and analyzing air in adsorbent materials to determine the chemical composition of collected samples, specifically a tube with a hydrophobic adsorbent element in a hybrid active and passive category for sampling volatile and semi-volatile compounds in environmental contamination investigations or for prospecting natural resource sources such as oil in its various forms and sampling processes.

[002] The management of contaminated areas is a sector that is more than 30 years old and during its evolution several tools were created to assist in the investigations necessary for management. Despite this, there is still a great shortage of economically viable and technically appropriate tools for environmental contamination assessments in contaminant scan mode. Tools for environmental investigation in scan mode require versatility of application and list of compounds, performance in different environmental conditions, such as very humid environments or even the presence of water that can affect the collection process and a reduced cost when compared to other techniques.

[003] An in-depth analysis of the existing technology by this inventor reveals a lack of volatile and semi-volatile compound samplers for hybrid active or passive sampling with quantitative analysis and solvent sample extraction, such as the Waterloo Membrane Sampler (WMS), which is a sampling device, a glassy container containing an adsorbent element with a lid and a vapor/gas permeable membrane, for quantitative assessment of volatile organic compounds by passive sampling only. The analysis is performed by a solvent sample preparation or by thermal desorption, processes which are followed by a gas chromatography analysis.

[004] Also, the US EPA TO-17 methodology discloses an analytical and sampling method disclosed by the US EPA for volatile organic compound analyses for quantitative evaluation by active-only sampling. Sampling is performed in tubes containing one or more adsorbent elements. The extraction is performed only by the thermal desorption methodology of the tube and then by gas chromatography analysis.

[005] Furthermore, the USEPA TO-3C methodology is an analytical and sampling method disclosed by US EPA for analysis of semi-volatile organic compounds for quantitative evaluation by active-only sampling. Sampling is performed in tubes containing adsorbent elements and also with a polyurethane filter. Preparation is done by extraction using solvents and the Soxhlet process. The analysis is performed by gas chromatography.

[006] Finally, the NIOSH methodology is an analytical method for occupational and sampling assessments described by NIOSH for analyses of a limited list of volatile organic compounds. Sampling is active and glass tubes containing coconut activated carbon are used. There is a major limitation in this case as coconut activated carbon is not hydrophobic and can be interfered with by moisture when applied to soil air sampling, typically with high moisture contents.

[007] In view of the gaps in the technique, the proposed invention is a sampling system comprising a tube (1) with a mixture of passive or active hybrid category adsorbent materials for sampling volatile and semi-volatile compounds for investigations of environmental contamination or for prospecting natural resource sources such as oil in its various forms and its sampling process.

[008] The invention is a tube (1) composed of metallic, glassy or polymeric material, rigid and inert to the substances or compounds to be sampled and analyzed. The outer surface of tube (1) has engravings of an arrow (3) indicating the sampling flow and tube (1) serial number (4) for traceability.

[009] Tube (1) has two polyethylene caps (5) for closure.

[010] Internally it is composed of a mixture of hydrophobic activated carbon spheres (2) of different sizes, from 0.4 to 0.8mm and surface area from 1000 to 1300m²/g that are fixed with the aid of metallic or vitreous fabrics or other inert materials (6) to the substances or compounds that will be sampled and analyzed. The set also has two stainless steel retaining springs (7) that prevent the movement of the internal elements inside said tube during active sampling.

[011] The invention may be better understood with the aid of the attached Figures.

[012] Figure 1 shows a front view of sample tube (1).

[013] Figure 2 reveals a side view of sample tube (1) without a cap.

[014] Figure 3 shows the internal composition of ~~the~~ tube (1).

[015] Figure 4 shows an expanded view of tube (1) and internal composition. For active sampling, in addition to sample tube (1), a syringe (8) coupled to a check valve system (9) is used.

[016] Figure 5 shows syringe (8) and check valve system (9).

[017] Sampling process is facilitated by avoiding errors during the process, as there is no need to disconnect sample tube (1) from the system. For active sampling, sample tube (1), syringe (8) and check valve system (9) are used, as below:

- i. Remove cap (5) from sample tube (1) section coincident with the direction of flow arrow (3);
- ii. Connect sample tube (1) to syringe (8) as shown in Figure 6;
- iii. Remove cap (5) still remaining from sample tube (1) and connect to the sampling point;

- iv. Pull and push syringe (8) plunger according to the required volume to be sampled. The check valve system (9) only allows airflow in the flows indicated by Figure 7, pulling the plunger, and Figure 8, pushing the plunger;
- v. Disconnect sample tube (1);
- vi. Replace both caps (5).

[018] Figure 6 shows tube (1) connected to syringe (8) and valve system (9).

[019] Figure 7 shows the flow by pulling syringe (9) plunger when air from the sampled point passes through sample tube (1).

[020] Figure 8 shows the flow pushing syringe (9) plunger when air is relieved by side check valve (9).

[021] For passive sampling only a specific sampling cap set (10) is used. It is a cap with a silicone tube (11) section, a rigid metallic or polymeric support (12) and a hydrophobic and oleophobic membrane (13) sandwiched between silicone tube (11) and rigid support (12), said sampling cap set (10) being made of polytetrafluoroethylene (PTFE) or acrylic copolymers, which allows free passage of the compounds and/or substances of interest and prevents the formation of barriers by the presence of water in liquid state in the catchment region of the compounds and/or substances of interest. In addition, membranes drastically reduce the passage of water in vapor form, which can cause analytical problems such as low sensitivity.

[022] Figure 9 illustrates sampling cap set (10) used for the passive sampling process.

[023] For passive sampling, sample tube (1) and sampling cap (10) are used, as below:

- A. Remove cap (5) from the sample tube (1) section opposite to the flow direction indicated by flow arrow (3);

B. Connect sample tube (1) to sampling cap set (10);

C. Position sample tube (1) at the location to be sampled. For example: Positioned within a building for indoor air assessment or ground burial for soil air assessment;

D. After the sampling period, normally 7-14 days, remove sample tube (1); and

E. Replace cap (5).

[024] Figure 10 shows the internal composition of sample tube (1) assembled for passive sampling.

[025] Figure 11 demonstrates a front view of sample tube (1) mounted for passive sampling.

[026] In summary, the new system-combined with the corresponding process is the only sampler designed for active or passive sampling. For this type of sampler, it is possible to analyze volatile compounds both-by thermal desorption and solvent extraction. Furthermore, extraction can be performed using different solvents and, therefore, allows the inclusion of analysis of semi-volatile compounds as well as multiple analyses including dilutions that are not suitable for the thermal desorption method and the adsorbent elements selected for the sampling of various organic compounds. The hydrophobicity of the adsorbent elements inhibits the interference of moisture by saturation and therefore does not generate false negatives.

[027] This invention is not limited to the representations commented or illustrated herein, and should be understood in its broad scope. Many modifications and other representations of the invention will come to the mind of one skilled in the art to which this invention belongs, having the benefit of the teaching presented in the previous descriptions and attached drawings. Furthermore, it is to be understood that the invention is not limited to the specific form disclosed, and that modifications and other forms are understood to be included within the scope of the appended claims.

Although specific terms are employed herein, they are used only in a generic and descriptive manner and not for the purpose of limitation.

CLAIMS

1. Sample tube (1) containing adsorbent elements for volatile and semi-volatile compound sampling, provided with serial number (4) and containing an adsorbent (2), **characterized by** comprising: a rigid, inert tube (1) the outer surface of same being engraved with an arrow (3); two caps, (5), and internally, as adsorbent, a mixture of hydrophobic activated carbon spheres (2) of different sizes, from 0.4 to 0.8 mm and surface area from 1000 to 1300m²/g, fixed in metallic and vitreous fabrics (6); and two-retaining springs (7), further comprising,
 - a) for active sampling, a syringe (8) and a check valve (9) to be attached to sample tube (1) after removal of cap (5) from sample tube (1) section in the direction indicated by flow arrow (3); and
 - b) for passive sampling, sampling cap set (10) comprising a silicone tube (11) section, a rigid support (12) and a hydrophobic and oleophobic membrane (13), to be attached to sample tube (1) after removal of cap (5) from sample tube (1) section opposite to the flow direction indicated by flow arrow (3).
2. Sample tube (1) according to claim 1b), **characterized in that** sampling cap set (10) comprises a silicone tube (11) section, a rigid support (12) and a hydrophobic, oleophobic membrane (13) sandwiched between silicone tube (11) and rigid support (12).
3. A process for active sampling of volatile and semi-volatile compounds to be carried out with the aid of sample tube according to claim 1, **characterized by** comprising the following steps:
 - i. Removing cap (5) from sample tube (1) section coincident with the direction indicated by flow arrow (3);
 - ii. Connecting sample tube (1) to syringe (8);
 - iii. Removing cap (5) still remaining from sample tube (1) and connecting same to the sampling point;
 - iv. Pulling and pushing syringe (8) plunger according to the required volume to

- be sampled, check valve system (9) only allowing airflow in the direction for pulling and pushing the plunger;
- v. Disconnecting sample tube (1); and
 - vi. Replacing both caps (5).
4. A process for passive sampling of volatile and semi-volatile compounds to be carried out with the aid of sample tube according to claim 1, **characterized by** comprising the following steps:
- i. Removing cap (5) from the sample tube (1) section opposite to the flow direction indicated by flow arrow (3);
 - ii. Connecting sample tube (1) to sampling cap set (10);
 - iii. Positioning sample tube (1) at the location to be sampled;
 - iv. After the sampling period, of 7-14 days, removing sample tube (1); and
 - v. Replacing cap (5).

INSTRUÇÕES:

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Nome do Beneficiário/CPF/CNPJ/Endereço

INSTITUTO NACIONAL DA PROPRIEDADE INDUST CPF/CNPJ: 42.521.088/0001-37

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Comprovante de pagamento de boleto

Dados da conta debitada / Pagador Final

Agência/conta: 0641/58387-7 CPF/CNPJ: 04.046.780/0001-61 Empresa: VSOL ANALISES AMBIENTAIS LTDA

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Nome do Beneficiário/CPF/CNPJ/Endereço

INSTITUTO NACIONAL DA PROPRIEDADE INDUST CPF/CNPJ: 42.521.088/0001-37

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