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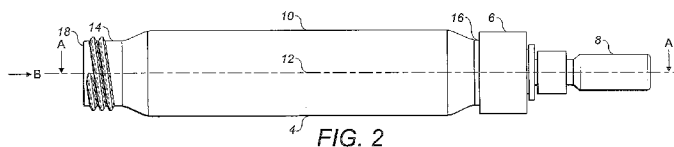
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(54) Title: METHOD AND SAMPLE HOLDING ASSEMBLY FOR USE IN SAMPLE PREPARATION



(57) Abstract: A method and sample holding assembly are described for use in the preparation of samples for analysis. In known techniques, following concentration of a sample using an evaporator, the concentrated solution is manually transferred to a smaller vial compatible with analysis apparatus using a pipette. This leads to loss of the sample and risks cross-contamination. Using the present method, a sample is concentrated directly into the vial. The method comprises the steps of collecting a sample in a concentration tube (4) which is open at one end and closed at the other, the closed end being selectively openable. A vial (8) is coupled to the open end, the tube is inverted, the closed end is opened, and then solvent is evaporated from the sample via the opened end until the concentrated sample is confined to the vial. A sample holding assembly for use in the present method is also described. It comprises a concentration tube (4), and an adaptor arrangement (6, 106) for coupling a vial (8) to the open end of the tube and defining a fluid path between the tube and the vial.



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Title: Method and sample holding assembly for use in sample preparation

Field of the Invention

The present invention relates to preparation of samples for analysis in which the samples comprise at least one solute dissolved in a solvent.

Background to the Invention

In order to prepare samples for analysis, it is often necessary to obtain a concentrated solution held within a vial compatible with the analysis apparatus. For example, a solvent extraction system may typically provide a sample consisting of a mixture of solutes dissolved in a solvent having a volume of around 40 ml or more. The sample then needs to be concentrated to a significantly smaller volume for further analysis. This concentration process may be carried out by a centrifugal evaporator, for example. The sample volume may typically be reduced to around 0.5 ml.

The concentrated solution is then transferred to a vial compatible with the analysis apparatus. This transfer is carried out manually using a pipette. This inevitably involves some loss of the concentrated sample and risks cross-contamination. Furthermore it is a slow process which needs to be carried out with great care.

It is known to provide an evaporation flask having a vial coupled to an opening in its base so that a sample can be concentrated directly into a vial (as in the SampleGenie products marketed by the present applicant). However, where the original sample is collected in apparatus employing relatively small containers, it is impractical to replace that container by a flask and vial combination due to the limited space available in the collection apparatus for the container.

Summary of the Invention

The present invention provides a method of preparing a sample for analysis, wherein the sample comprises at least one solute in a solvent, comprising the steps of:

providing the sample in a concentration tube which is open at one end and closed at the other, the closed end being selectively openable;

coupling a vial to the one end;

inverting the tube;

opening the other end of the tube; and

evaporating solvent from the sample via the other end of the tube, until the concentrated sample is confined to the vial.

Accordingly, the sample is initially collected in the concentration tube, the dimensions of the concentration tube being compatible with the system creating the sample. The sample in the concentration tube can then be readily and reliably prepared for concentration directly into a vial by coupling the vial to the open end of the tube and inverting the tube. The assembly comprising the concentration tube and vial can be loaded into an evaporator, allowing the sample to be concentrated into the vial for further processing.

Thus, the vial may be decoupled from the tube after the evaporation step and loaded into analysis apparatus, such as chromatographic analysis apparatus. More particularly, the analysis apparatus may be a gas chromatography system and the vial a gas chromatography vial compatible with such a system.

The present invention further provides a sample holding assembly comprising:

a sample holding assembly comprising:

a concentration tube which is open at one end and closed at the other, the closed end being selectively openable; and

an adaptor arrangement for coupling the vial to the open end of the tube and defining a fluid path therebetween.

The adaptor arrangement may advantageously comprise an adaptor having a tube end for coupling to the tube and a vial end for coupling to a vial, and a container (or other retaining device) for receiving a vial, the container being arranged to couple with the vial end of the adaptor such that the open end of the vial is held in fluid communication with the fluid path through the adaptor.

More particularly, the container and adaptor are coupled together by complementary screw threads, and the adaptor arrangement includes a seal configured such that as the container is screwed onto the adaptor, the open end of a vial held in the container is urged against the seal.

Preferably, the other, closed end of the concentration tube is closed by a removable cap, or a closure having a fluid path therethrough which is selectively openable to allow evaporated solvent to escape via that end of the tube.

In a preferred embodiment, the removable cap and tube are coupled together by complementary screw threads. Similarly, the adaptor and tube may be coupled together by complementary screw threads. In particular, the screw threads at each end of the tube may be of substantially the same pitch.

Brief description of the Drawings

Sample holding assemblies embodying the invention will now be described by way of example with reference to the accompanying drawings, wherein:

Figure 1 is a cross-sectional side view of a concentration tube and cap;

Figure 2 is a side view of the concentration tube with a vial coupled to one end;

Figure 3 is an end view in direction B of the assembly of Figure 2;

Figure 4 is a cross-sectional side view along line A-A of the assembly of Figure 2;
and

Figure 5 is a cross-sectional side view of another embodiment of a sample holding assembly.

Detailed description of the Drawings

Figure 1 shows a concentration tube 4 and an associated end closure in the form of cap 3. A fluid-tight seal between the tube and cap is provided in the form of a seal 5. The seal is formed of an inert deformable material, such as PTFE for example.

The sample holding assembly 2 shown in Figures 2 to 4 comprises a concentration tube 4, and an adaptor 6, and a concentration vial 8 (a cap 3 is omitted from these Figures).

The concentration tube 4 has a cylindrical mid-section and a central, longitudinal axis 12. It also includes cylindrical end sections 14 and 16 having a smaller diameter than the mid-section. For example, in a concentration tube having a volume of around 40 ml, the mid-section has a diameter of around 27.5 mm, and a length of around 140 mm, with the diameter of the end sections 14, 16 being around 17 mm. Each end section defines a respective opening 18,20 at opposite ends of the tube 4. A screw thread 22,24 is formed on the outer circumferential surface of each end section 14,16 respectively. The threads are of equal pitch, but different threads could instead be provided at each end. The tube is formed of borosilicate glass, for example.

The dimensions of the concentration tube are such that it is compatible with an extraction system used to collect the sample to be analysed. An example of such an extraction system is the Accelerated Solvent Extraction (ASE) system marketed by Dionex.

The vial 8 may be a gas chromatography vial. Common dimensions for such a vial are a diameter of 12 mm and a length of 32 mm.

Vial 8 is coupled to the opening 20 of the concentration tube by adaptor 6. As can be seen in Figure 4, the adaptor consists of a connector 40 which defines a fluid path 42 along its central axis to allow fluid to flow between the concentration tube and the vial. Connector 40 is held against the open end 20 of the concentration tube by a threaded coupling 44 which engages the thread 24 on the tube. A second coupling 46 holds the connector 40 against the open end of the vial 8 by screwing onto a thread defined around the open neck of the vial. The components of the adaptor may be formed of polypropylene, PTFE, or another inert polymer for example.

Figure 5 shows a sample holding assembly 102 similar to that of Figures 2 to 4. It illustrates an alternative way to fasten the vial 8 to the concentration tube 4.

Vial 8 is coupled to the opening 20 of concentration tube 4 by an adaptor 106. The adaptor defines a fluid path 142 along its central axis between the vial and the tube. It includes a screw thread 152 on an inner surface at one end for connection to thread 24 on the tube, and a further screw thread 154 on an outer surface at the other end.

A container 156 in the form of a cylinder closed at one end is configured to receive and contain vial 8 and closely fit around its base. An inner surface 162 of the closed end of the container is profiled so as to complement the circumferential region at the base of the vial and retain it in a central location within the container. The open end of the container defines a thread 158 on its inner surface for engagement with thread 154 of the adaptor 106. An O-ring seal 160 is held between the adaptor 106 and the open end of the vial 8.

As container 156 (with vial 8 held inside) is screwed onto adaptor 106, the vial is urged against the seal 160 so as to form a fluid-tight seal between vial and adaptor.

The adaptor and container configuration of Figure 5 is particularly beneficial when it is desirable to insulate the vial to some extent during the concentration process. This may be the case for example when using a condensing evaporator (of the form described in a United Kingdom patent application published under no. 2436075 and filed by the present applicant). The insulation provided by the container serves to

slow down the evaporation process allowing close control of the end point to avoid drying out the sample completely.

Use of a sample holding assembly of the form shown in the drawings in a method of preparing a sample for analysis will now be described. Initially, a concentration tube 4 with one end closed by a cap 3 is inserted into the extraction system and the sample collected in the tube. The tube containing the sample is removed from the extraction system and an adaptor 6 and vial 8 coupled to the open end of the tube. The tube is then inverted so that the sample flows into the concentration vial 8, with the remainder in the end of the tube connected to the vial.

The cap 3 is then removed, before loading the assembly into a concentrator such as a centrifugal evaporator. In the concentrator, solvent is evaporated from the sample via the open end 18 of the tube such that the sample is concentrated directly into the vial 8.

It would generally be impractical to provide a concentration tube, vial and adaptor for loading directly into an extraction system as this would lead to unacceptable reduction in the volume of sample that may be collected due to the limited space available. According to the present invention, the concentration tube alone is loaded into the extraction system whilst still allowing for reliable and efficient production of a concentrated sample in a separate vial.

Claims

1. A method of preparing a sample for analysis, wherein the sample comprises at least one solute in a solvent, comprising the steps of:
 - providing the sample in a concentration tube which is open at one end and closed at the other, the closed end being selectively openable;
 - coupling a vial to the one end;
 - inverting the tube;
 - opening the other end of the tube; and
 - evaporating solvent from the sample via the other end of the tube, until the concentrated sample is confined to the vial.
2. A method of claim 1, including the steps of decoupling the vial from the tube after the evaporation step, and loading the vial into analysis apparatus.
3. A method of claim 2, wherein the analysis apparatus is chromatographic analysis apparatus.
4. A method of any preceding claim, wherein the vial is a gas chromatography vial.
5. A sample holding assembly comprising:
 - a concentration tube which is open at one end and closed at the other, the closed end being selectively openable; and
 - an adaptor arrangement for coupling a vial to the open end of the tube and defining a fluid path therebetween.
6. An assembly of claim 5, wherein the adaptor arrangement comprises an adaptor having a tube end for coupling to the tube and a vial end for coupling to a vial, and a container for receiving a vial, the container being arranged to couple with the vial end of the adaptor such that the open end of the vial is held in fluid communication with the fluid path through the adaptor arrangement.

7. An assembly of claim 6, wherein the container and adaptor are coupled together by complementary screw threads, and the adaptor arrangement includes a seal configured such that as the container is screwed onto the adaptor, the open end of a vial held in the container is urged against the seal.
8. An assembly of any of claims 5 to 7, wherein the other end of the tube is closed by a removable cap.
9. An assembly of claim 8, wherein the removable cap and tube are coupled together by complementary screw threads.
10. An assembly of any of claims 5 to 9, wherein the adaptor and tube are coupled together by complementary screw threads.
11. An assembly of claims 9 and 10, wherein the screw threads at each end of the tube are of substantially the same pitch.
12. An assembly of any of claims 5 to 11, wherein the adaptor arrangement is configured to receive a gas chromatography vial.
13. An assembly of any of claims 5 to 12 for use in a method of any of claims 1 to 4.
14. A method of preparing a sample substantially as described herein with reference to the Drawings.
15. A sample holding assembly substantially as described herein with reference to the Drawings.

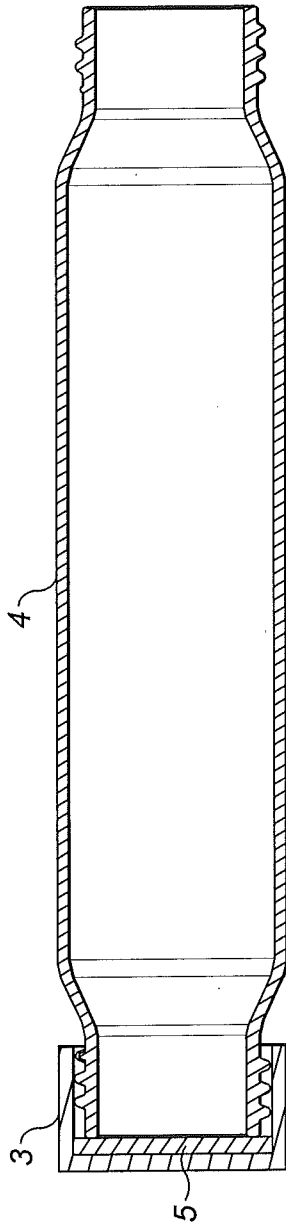


FIG. 1

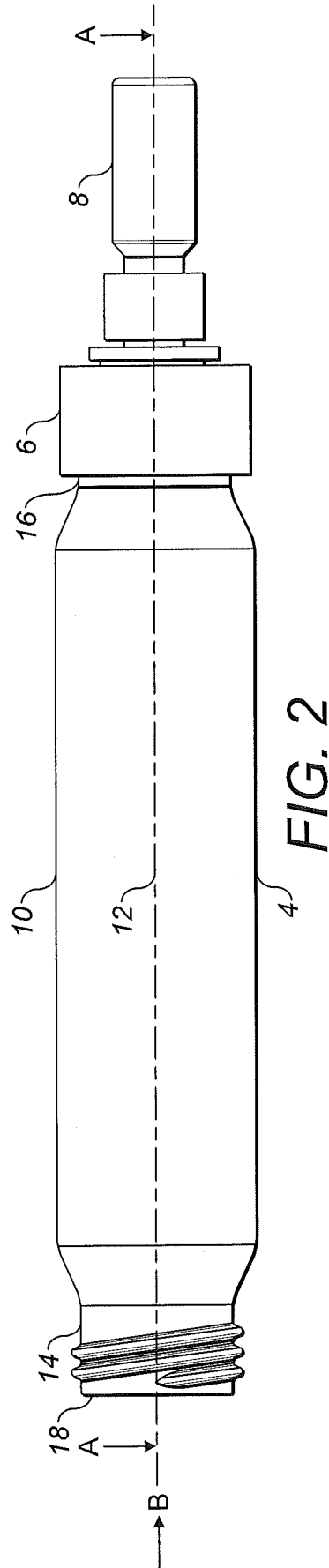


FIG. 2

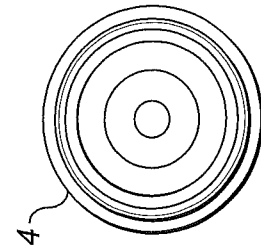


FIG. 3

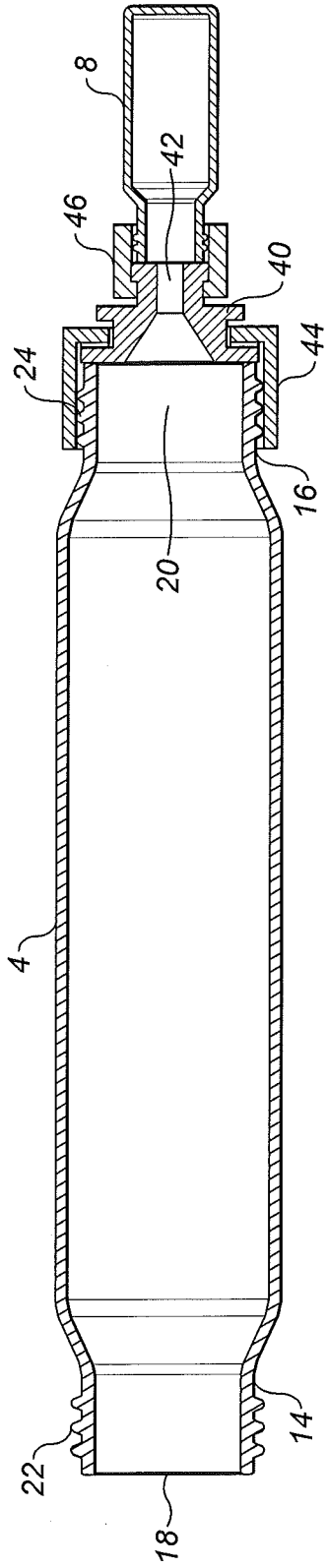


FIG. 4

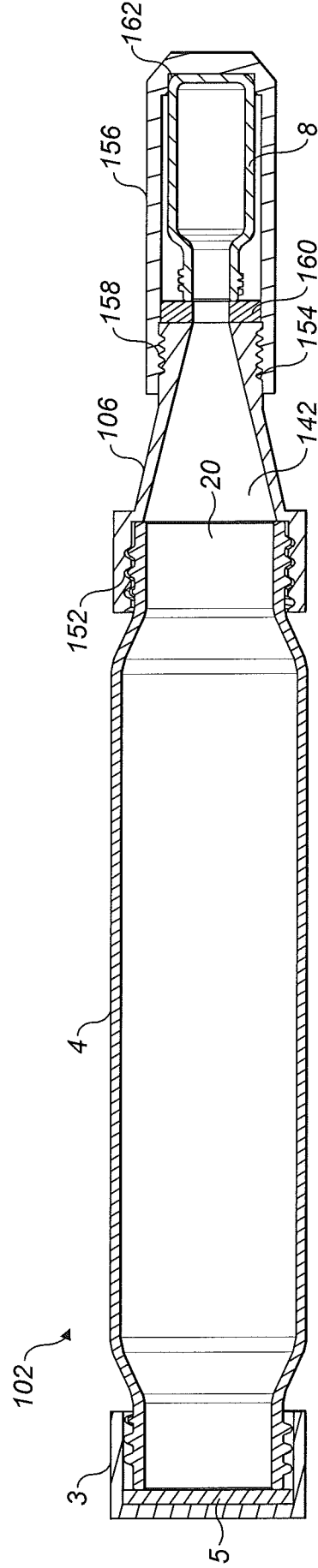


FIG. 5

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER INV. B01D1/22 B04B5/04 G01N1/40 ADD.		
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) B01D B04B 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		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2007/130991 A1 (HORIZON TECHNOLOGY INC [US]; JOHNSON ROBERT A [US]; SCHEUFELE STEVE [U] 15 November 2007 (2007-11-15) pages 4,5; figures 1-4 -----	1-15
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<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C.		
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Date of the actual completion of the international search <div style="text-align: center; font-weight: bold;">25 May 2010</div>	Date of mailing of the international search report <div style="text-align: center; font-weight: bold;">08/06/2010</div>	
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer <div style="text-align: center; font-weight: bold;">Cantalapiedra, Igor</div>	

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Information on patent family members

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