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(54) Title: A DOSING APPARATUS ASSEMBLY FOR REACTIVE CHEMICALS

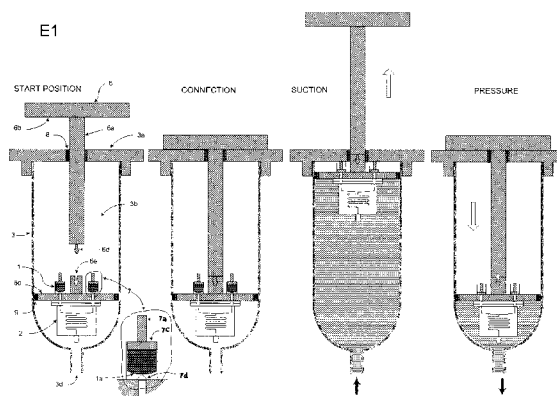


Figure 1

(57) Abstract: The present invention relates a batch operated dosing apparatus assembly for producing an active chemical product. The apparatus comprises i. at least two sealed reactant containers (1) for providing at least two liquid reactants for a chemical reaction taking place inside said dosing apparatus assembly (E) and resulting in said active chemical product, ii. at least one microstructured device (2) providing a reaction space for said chemical reaction with efficient mixing for said reactants, whereby the inlet of at least one device is adapted to receive the liquid reactants from said containers (1), iii. a casing (3) functionally connected to said sealed reactant containers (1) and said microstructured device(s) (2), and optionally being capable of enclosing liquid solvent which is able to dissolve or dilute said active chemical product and iv. means (20) for launching said reaction and/or carrying said liquid solvent in-side said casing (3).

A dosing apparatus assembly for reactive chemicals

The present invention relates to batch operated dosing apparatus assembly suitable for syntheses of active chemical products. Furthermore, the invention relates to a method for using said apparatus.

Background

Chemical compounds having at least one O-O group consisting of two linked oxygen atoms are generally referred to as peroxygen compounds or peroxygens. Peroxygens are a family of products comprising hydrogen peroxide, percarboxylic salts or percarboxylic acids. The main source for the oxidation behaviour is hydrogen peroxide that is a strong oxidising agent and used as such mainly for bleaching of pulp and de-inked recycled paper. Other uses include industrial applications, such as textile bleaching and disinfectant in aseptic packaging, as well as personal and domestic applications such as hair bleaching, wound disinfection and contact lens maintenance. Percarboxylic salts such as sodium percarbonate are key ingredients in detergents for textile garments and for dish cleaning. As a bleaching agent, it removes stains and has sanitizing properties. Percarboxylic acids or peroxy acids are used as disinfectants and intermediates for chemical synthesis.

Percarboxylic acids are formed by contacting aqueous hydrogen peroxide and aqueous carboxylic acid in liquid phase. Performic acid, peracetic acid and perpropionic acid can be mentioned as industrially important percarboxylic acids. Generally, part of the original reagents and reaction products form an equilibrium mixture.

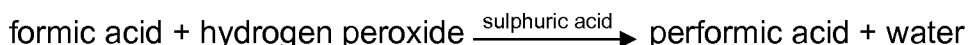
However, the equilibrium mixtures of percarboxylic acids and in particular performic acid may be rather unstable, reactive and/or hazardous in handling and in storage and have sometimes a relative short shelf life. However, these compounds are very useful and effective in various fields of technology such as disinfection due to their advantageous properties. Performic acid is the simplest and most powerful percarboxylic acid. Formed from the reaction of hydrogen peroxide and formic acid, it reacts more rapidly and powerfully than peracetic acid before breaking down to water and carbon dioxide. Performic acid is an environmentally friendly oxidising biocide for various disinfection applications. The application areas involve microbial growth control and cleaning of surfaces in larger industrial scale such as municipal or industrial waste water purification, or for circulation of

process waters in pulp and paper industry. These reagents are most applicable for example in hospitals, dental surgeries, kitchens and bathrooms to kill infectious organisms.

5 In WO9420424 performic acid has successfully been applied to microbial control for horticulture. It discloses a method for preventing and combating harmful micro-organisms such as fungi, viruses, bacteria, yeasts and algae in water circulation systems and appliance to be used therewith. A dilute performic acid solution is fed into the nutrient feed solution or drain water and it is shown to prevent the growth of algae in the piping thus hampering the plugging tendency. When compared to
10 peracetic acid equilibrium solution the disinfection power of performic acid is found clearly higher due to its superior reactivity towards microbes. The disinfection power of formic acid in the absence of hydrogen peroxide has been proven to be negligible compared to percarboxylic acids.

15 In the market, there are several products containing performic acid such as Resi-clean® or Hyproform®, for example. These products are strong oxidizers with notable antimicrobial properties. Performic acid solutions are highly reactive. If performic acid solutions are contacted with impurities such as zinc dust, lead dioxide or sodium azide they may react violently and decompose. Performic acid typically decomposes as such into carbon dioxide and water within a few hours at ambient
20 temperature and pressure.

Performic acid is formed by reacting aqueous formic acid with aqueous hydrogen peroxide through an exothermic reaction in the presence of a strong mineral acid catalyst, such as sulphuric acid:



25 The reaction above achieves reaction equilibrium in which all the components are present in the liquid mixture.

Due to instability and high reactivity of performic acid, the equilibrium solutions are highly unstable. Therefore, performic acid solutions should be prepared in situ preferably at the point of use or directly before use depending on the properties of
30 the reactants and reaction products.

One solution for controlling and managing exothermic oxidation reactions of organic compounds creating rapid or even explosive release of heat of reaction is disclosed in CA2247662. The key issue in this document is to perform the reaction

in a temperature controlled manner using a reactor having sufficiently high heat transfer efficiency. This problem is solved by using a downstream microreactor where to at least two material streams are fed after mixing them with a jet mixer. This microreactor has reaction channels and cooling channels with size dimensions less than a millimetre, and it is serially subdivided into two microreactor stages which reaction channel cross sectional areas increase stepwise in the flow direction. In addition to the microreactor the manufacturing equipment comprises separate vessels for reactants, means for transporting the reactants into the reactor such as pumps, valves and filters, a jet mixer for premixing the solution and several pipelines. The system described is meant for large capacities, tens of kilograms per hour. It requires an electricity source e.g. for pumps and mixers, and a control system that has to be accurate in order to achieve the quality required. On-site installation is necessary before taking it into use. Besides, the system is not closed with through holes for the mixers. The cost for such a system can be quite high.

US2002/0192118 discloses microreactor coating materials selected from silicon dioxide, silicon nitride and/or aluminium oxide to avoid or reduce unwanted side reaction between the reactor material and reactants. The formation of performic acid by the action of hydrogen peroxide on formic acid is used as a reactive probe set for the coatings. It is not disclosed how the components are fed into the micro-reaction system and what kind of product composition is formed. Furthermore, the microreactor system or flow characteristics of the reactants are not disclosed.

Microstructured or microchannel reactors or devices are generally called microreactors that can also be defined as miniaturized reaction systems. The lateral dimensions of channels in microreactors range typically from 50 microns or even less up to 2 millimeters. Companies constructing microreactors provide microstructured mixers, heat exchangers and reaction modules for gas and liquid phase reactions. These microstructured systems consist usually of microstructured plates including flowing channels for fluids. There are also microstructured separation units and gas-liquid reactors available. Reactor modules for liquid phase reactions and gas-liquid reactions consist usually of both reaction and heat exchange channels. Microstructured devices dedicated for multiphase operations especially with solids are however rare.

Microreactor technology is most suitable for chemical reactions. The reduced dimensions in microreactors make them pertinent for various reactions that need good transport properties for heat and mass transfer. An other important feature of

microreactors is their high surface-to-volume -ratio. This is indispensable especially for reactions that require effective heat transfer. In stirred reactors reactions usually take place in agitated tanks, the reaction rate is often limited by low heat transfer capacity because of poor surface-to-volume -ratio, and therefore, heat cannot be introduced or removed fast enough. In a microreactor, the rate of the process is not limited by heat transfer capability but rather by the intrinsic reaction kinetics. It is even possible to carry out fast or instantaneous and highly endothermic or exothermic reactions in isothermal conditions and under an accurate temperature control in microreactors. Isothermal conditions and short reaction times result in improved yields and selectivities compared to conventional reactor technology. Small dimensions of reaction channels entail short diffusion paths for components, hence the mixing is efficient even if the flow is laminar. Accordingly, efficient mass and heat transfer accomplished by microreactors provide more uniform concentration and temperature profiles that in turn decrease by-product formation and therefore the selectivities of the reactions improve towards the desired products.

Microreactor technology is also practical for continuous production of chemicals with continuous flow reactors. The small reactor volume together with efficient heat transfer properties improves also safety. Intrinsically safe aspects, such as small dimensions, small amounts of reagents and good controllability make microreactors applicable especially for hazardous reactions. Microreactors offer also other advantages over conventional scale reactors, such as reliability, scalability and on-site or on-demand production.

Microstructured devices have already several production scale applications, particularly in fine and pharmaceutical chemical industry. Generally, if the reaction is fast and heat evolution considerably high, formation of by-products is likely and efficient heat transfer is needed. Then, in order to gain sufficient productivity, microreactor technology may be the way to intensify the process.

Microreactors can also be used for heterogeneous catalytic reactions. Thin catalyst layers – even in nanometer scale – may be used in microreactors, which minimizes the diffusion resistance and improves the overall effectiveness of the catalyst. This suppression of diffusion resistance results in process intensification by substantial minimization of the processing equipment.

There has not been available a suitable apparatus for managing reactive or hazardous chemicals which is portable and easily operated without any particular

knowledge either in the chemical properties of compounds or equipment technology.

The object of the present invention is to provide a one-piece portable apparatus suitable for in situ synthesis of active chemical products, especially peroxygen products, specifically such as performic acid, to be used for dosing of the chemical product at the point-of-use.

The other objectives of the present invention are to provide a user-friendly, easily operated, efficient and economical apparatus and a method suitable for storing and synthesising chemical compounds which are difficult to handle or even hazardous.

Summary of the invention

The present invention discloses a batch operated dosing apparatus assembly for providing an active chemical product as defined by claim 1. Furthermore, a method for preparation of said chemical product using this dosing apparatus assembly is provided as set out in claim 13. The invention further includes use of the inventive apparatus assembly and the associated method as depicted by claim 15.

The present invention expands the use of microstructured devices such as micro-reactors from industrial environment wherein they are typically used as one part of larger equipment into ready-to-use dosing apparatus assembly for the consumer market. This apparatus assembly is readily usable and stored as such and it is easy to handle and use wherever and whenever needed. Reactive or even hazardous chemicals may thus be manufactured in-situ and dosed at the point of use. Moreover, the apparatus assembly is maintenance free and may be reused or recycled if so preferred.

The inventors found out that for example performic acid could be safely generated by enclosing precursor reactant chemicals separately into sealed containers such as capsules, located inside this portable dosing apparatus.

The apparatus assembly of the present invention does not necessarily require any installation or instrumentation to take it into use. Neither connections to electrical power sources nor any specific operational skills are necessary. It is portable, self-sustained, light-weighted and compact one-piece apparatus that is easy to carry and use at the specific application when needed. It provides preset dosing of an active chemical solution and there are neither fluctuations in the quality of the end

product nor shelf life issues associated when operated by different users at irregular periods of time.

Figure 1 presents one example of the dosing apparatus assembly of syringe type for dilute product solutions.

- 5 Figure 2 presents one example of a dosing apparatus assembly with a spraying bottle.

Figure 3 presents one example of a dosing apparatus assembly of syringe type for concentrated product solutions.

A detailed description of the invention

- 10 The dosing apparatus assembly is a one-piece compact set of intergrated parts required for synthesising the desired active chemical product and providing it to the point of use safely. "One-piece compact set of intergrated parts" means here, that the parts of the assembly are functionally connected together and at least at the time of use they are also contacted with each other.
- 15 This batch operated dosing apparatus assembly comprises the following parts:
- i. at least two sealed reactant containers for providing at least two liquid reactants for a chemical reaction taking place inside said dosing apparatus assembly and resulting in said active chemical product,
 - 20 ii. at least one microstructured device providing a reaction space for said chemical reaction with efficient mixing for said reactants, whereby the inlet of at least one device is adapted to receive the liquid reactants from said containers (1),
 - 25 iii. a casing functionally connected to said sealed reactant containers (1) and said microstructured device(s), and optionally being capable of enclosing liquid solvent which is able to dissolve or dilute said active chemical product and
 - iv. means for launching said reaction and carrying said liquid solvent inside said casing.

The above containers provide the reactants for the required chemical reaction taking place inside the dosing apparatus assembly to produce the desired active chemical product. These containers are adapted to be connected to an inlet of at least one microstructured device via suitable transportation means for liquids. This device provides a reaction space for said chemical reaction with efficient mixing for said reactants due to the microstructure for initiating and possibly also completing the synthesis of the desired active chemical product. The casing is functionally connected to the sealed reactant containers and the microstructured device(s). Preferably, this casing encloses the reactant containers and is capable of enclosing inside liquid solvent which is able to dissolve or dilute said active chemical product. This casing may be composed of two or more parts that can be readily disassembled.

The target of this kind of dosing apparatus assembly is to provide a chemical reaction by contacting two or more reactants that react together. This chemical reaction is selected from the group of an oxidation reaction, a reduction reaction, a neutralisation reaction, a peroxy reaction or a catalytic reaction. The number of reactant containers may be increased depending on the nature of the reaction that provide the active chemical product desired.

Active chemical product is produced by using the above mentioned dosing apparatus assembly, which method comprises at least the following steps:

- taking liquid solvent inside the casing by means,
 - breaking the containers partly or completely for freeing the reactants,
 - mixing the reactants in the microstructured device for producing an active chemical product.
- The above method may further comprises a step for dissolving or diluting the active chemical product into liquid solvent for achieving a product solution and discarding said product solution from the dosing apparatus assembly by means such as a piston.

By the term "active chemical product" is meant a product of a chemical reaction which is a substance, functional at the intended application. This product may be a reactive chemical, a hazardous chemical, a chemical that undergoes an exothermic process when formed/produced or e.g. diluted with water, a chemical that re-

acts further in ambient use conditions or an unstable chemical with a short shelf life.

The active chemical product produced is in the form of an liquid solution, preferably an aqueous solution product of a neutralization or peroxy reaction. More preferably the active chemical product is in the form of a peroxygen compound, most preferably a percarboxylic acid, such as a performic acid.

Performic acid is the most preferred active chemical product. It is preferably obtained as an aqueous solution of a concentration less than 15% by weight, preferably 100 ppm – 14% by weight, more preferably 500 ppm – 14% by weight. Reactants commonly known in the state-of-the-art are used for the reactions. Performic acid is preferably prepared by reacting hydrogen peroxide solution with formic acid solution in the presence of a sulphuric acid catalyst. More preferably, one of the reactants is an aqueous solution of formic acid and/or one of the reactants is an aqueous solution of hydrogen peroxide. Most preferably, one of the reactants is an aqueous solution of formic acid of 10–85% by weight and one of the reactants is an aqueous solution of hydrogen peroxide of 5–75% by weight. Depending on the desired concentration of the active chemical product, preferably performic acid, the proportion of water and the size of the containers may be varied.

In neutralisation reactions, aqueous salt solutions are produced. Preferably, carboxylate salt solutions are produced. Even more preferred is the production of ammonium and alkali or alkali earth metal carboxylate solutions. Ammonium, sodium or potassium salts of carboxylates are especially preferred. Even more preferred is the synthesis of ammonium, sodium or potassium formates.

The reactants to be used inside the reactant containers include liquids with viscosity preferably less than 10000 cP, more preferably less than 1000 cP, most preferably less than 100 cP.

In one embodiment the dosing apparatus assembly comprises at least two sealed reactant containers, the content of at least one of which comprises reactant(s) for the desired chemical reaction and at least one of which comprises an auxiliary agent, diluting fluid or preferably a catalytically active material.

In another embodiment the dosing apparatus assembly comprises at least three sealed reactant containers containing reactants one of which comprises formic acid, one of which comprises hydrogen peroxide and one of which comprises an inorganic mineral acid, preferably sulphuric acid, more preferably concentrated

sulphuric acid as catalytically active material. Most preferably, formic acid is an aqueous solution of a concentration from 10–85% by weight and hydrogen peroxide is an aqueous solution of a concentration from 5–75% by weight and sulphuric acid is an aqueous solution of a concentration from 10 to 98% by weight.

- 5 By the term "efficient mixing" is meant that the mixing time of reactants is short, preferably less than 1 second, and that the reactants are contacted fast making an immediate reaction possible. The reactants are contacted efficiently producing a homogenized composition with minor concentration deviations. The known advantages associated with microstructured devices such as microreactors apply.
- 10 By the term "sealed" is meant a closed system which has no material exchange with the surroundings when not in operation. This system preserves the reactants inside the containers when in storage or not taken into use, thus preventing any leakage or reactions with each other or ambient. However, this seal is suitable for being punctured or otherwise breakable at the time of use by the user.
- 15 The apparatus assembly is preferably readily portable. By the term "portable" is meant that the weight and form of the apparatus assembly is such that an average person is able to carry it and manages the use of the apparatus assembly without any further assistance.

- 20 The operability of the apparatus is determined by the batch capacity thereof which is 50–5000 ml, preferably 100–2000 ml, more preferably 100–1000 ml, most preferably 100–500 ml, such as 100–300 ml.

- 25 The weight and thus the operativity is further dependent on the hardware weight that is determined by construction material selected and size of the containers, casing, discharge means and the microstructured device. Naturally, the number and size of the reactant containers are limited by the dimensions and weight requirement of the apparatus assembly. Advantageously, the apparatus assembly is to be operated without any additional tools, preferably single-handed and may thus not weigh too much. Preferably, the dimensions of the apparatus assembly are less than one meter in length and less than 10 kg in weight, more preferably less than 1 kg, most preferably equal to standard syringe construction or regular commercial consumer spray bottles, such as length less than 300 mm and weight less than 500 g. The volumes of the reactant containers for reactants varies from 10 microliters to 1 liter, preferably from 100 microliters to 200 milliliters.
- 30

There may be an elevated pressure inside the reactant container(s) to enhance the flowability. The release of reactant to microstructured device is provided through releasing the overpressure by puncturing the seal.

5 Preferably, the reactant container comprises a puncturable seal. More preferably, the containers are sealed by a breakable foil (for example metal foil or laminated foil), film (for example polymer film) or diaphragm that can be readily punctured to let the chemical to be discharged.

10 The dimensions of the microstructured device inside the apparatus are determined by the size of the hydraulic channels required for the reaction and the thickness of the walls used. According to a preferred embodiment of the invention the microstructured device comprises channels having a hydraulic diameter from 10 micrometers to 2 millimeters, preferably from 100 micrometers to 1 millimeters. These channels are preferable multilayered channels in a multilayered device system.

15 The dosing apparatus assembly includes one or more microstructured devices depending on the application and desired product concentrations required. The essential performance criteria for the microstructured device is to mix the materials of the reactant containers as efficiently as possible.

20 A microstructured device for mixing fluids has preferably a backbone that comprises a plurality of primary and secondary micro-channel layers. Each layer comprises at least one input port and at least one output port connected by a micro-channel. The primary micro-channel layers are built up to receive the first fluid and the secondary micro-channel layers are built up to receive the second fluid. Within the layered device, the fluids traversing in the microchannel layers intermingle and are thus efficiently mixed.

25 Due to small flow channels the general restrictions of mixing, mass and heat transfer can be eliminated in microstructured devices. The fluid film in channels can be only tens of micrometers and therefore the mixing times can be microseconds, preferably nanoseconds. Such mixing times are not reachable in conventional mixing equipment. As the diameters of mixing channels can be as low as 10 μm the heat transfer area per volume may be as high as 10 000 – 50 000 m^2/m^3 whereas
30 in conventional laboratory or production equipment heat transfer areas of 100–2000 m^2/m^3 can be reached. Normal values for the overall heat transfer coefficient in conventional devices are 2500 $\text{W}/\text{m}^2/\text{K}$ at maximum while 10-fold values can be reached with microstructured devices. Microstructured devices serve better also in

two-phase systems as the interphasial surface areas may be as high as $30\,000\text{ m}^2/\text{m}^3$ whereas in conventional bubble columns values of $100\text{ m}^2/\text{m}^3$ can be reached. A microstructured device generates good contact for the reactants for mixing and reacting, and subsequently good conversion is obtained for the chemical product.

The efficient mixing provided is due to capillary flow in small diameter microchannels. The extent of mixing achieved by this arrangement is remarkably good compared to conventional mixing equipment such as conventional static mixer, stirred tank reactor or tubular flow reactor.

The reaction that is initiated after mixing the reactants and which is taking place inside a microstructured device is optionally complemented by connecting the microstructured device output to the means for completing said chemical reaction in case the reaction is not completed instantaneously. The need for completion of the reaction depends on the kinetics of the reaction. The apparatus assembly of the present invention preferably further includes means for completing said chemical reaction, preferably an auxiliary space to increase the residence time of the reaction mixture inside the apparatus assembly and to complete the reaction into the desired chemical product with good yield. More preferably, the auxiliary space is a tubing which is directly connected to the outlet of the casing.

The reactant containers, the microstructured device and the means for transporting the fluids to microstructured device are enclosed or connected to the casing of dosing apparatus assembly. Thus, the dosing apparatus assembly is preferably in a form of a spray bottle or a syringe or a grease pump. Locating the reactant containers inside the casing of dosing apparatus assembly ensures that e.g. in case of leakage the escaping fluid is trapped inside the apparatus. More preferably, the casing of apparatus comprises an inner wall and an outer wall. This space can be used to enable cooling or complete the reaction. The free space inside the casing is suitable for being filled with dilution liquid, preferably with solvent. This solvent will be able to dissolve the reactants and/or the reaction products. Preferably, the solvent is water.

As the apparatus assembly according to the invention is especially useful for handling precursor chemical reactants and/or end products which are hazardous or irritating. For this purpose it needs to be a sealed system. The reactant containers are designed to keep the reactants apart from each other and the solvent until the

reactants and thus the reaction is launched. Furthermore, the reactant containers are designed to keep the reactants from contact to surroundings.

The means for launching the reaction and/or transporting or carrying the solvent inside the casing comprise at least a device for breaking or decomposing the containers completely or partly. This device can be formed from several parts and is preferably made of punchers that break the puncturable seal of containers against a proper spike or the like formation and allow or force the reactants to flow into the microstructured device or microreactor to be contacted, mixed and reacted. In this way the chemical product is brought directly to use and also the enthalpy provided by reaction or dilution and transferred by convection may be utilized. The means for launching the reaction i.e. means for initiating discharge of reactant may also contain means for transporting or carrying the active chemical product out of the apparatus through an outlet such as a nozzle or the like. Preferably, an outlet nozzle has an aperture which is adjustable and may be fitted closely to a receiving device such as an inlet nozzle of a distribution device which ensures that the active chemical product is applied only to the point where it is needed.

According to a preferred embodiment the means for launching the reaction and/or diluting liquid solvent inside the casing include a transporting means, preferably a hand-powered device whereby the reactant solution is preferably forced through the outlet of the apparatus by back-pressure built up by a piston or crank arm.

An example of an apparatus assembly E operating as described above is shown in figure 1. This figure depicts a multipiston syringe E; E1. This syringe consists of two sealed reactant containers 1 (only one marked), a microstructured device 2, a casing 3 encasing the containers 1 and the microstructured device in a free space 3b inside the casing 3. As means 20 for launching the reaction and carrying or transporting liquid solvent, a transporting device 20; 20a that is a primary piston 6, is provided. Piston 6 is composed of a plunger handle 6b, a plunger shaft 6a and a detached piston head 6c that is connectable to the microstructured device 2. The reactant containers 1 are also connectable to the piston head 6c. The plunger shaft 6a has a collet 6d that can be connected to the piston head 6c via a collet nut 6e. The bottom of reactant container 1 is made of foil or diaphragm 1a that can be punctured to release the liquid out from the container 1. Means 20 for launching the reaction includes also the puncturizing device 20; 20b that comprises secondary piston system 7 connected to each container 1. These secondary pistons 7 are mounted above the container 1. The piston system 7 comprises a plunger shaft 7a and a piston head 7c. Below each container there is a prong 7d belonging

also to the mentioned puncturizing device 20; 20b that punctures the diaphragm 1a when the plunger shaft 7a of the piston 7 is pressed against the top wall 3a of the casing 3. When the prong 7d pierces the diaphragm 1a, the reactant container 1 can be emptied as the plunger shaft 7a is pressed down. When the syringe is
5 taken into use the original position is the START POSITION. Subsequently, the collet 6d is pressed to collet nut 6e, in making the CONNECTION the piston head 6c is lifted to perform the SUCTION and diluting liquid is sucked to the space 3b inside the casing 3 through an aperture 3c of the bottom of the syringe casing. When plunger shafts 7a reach the top wall or cover 3a of the casing 3 the dia-
10 phragms 1a are punctured by the action of pistons 7 on containers 1 and liquid of the containers 1 enters into the microstructured device 2 where the reactants are rapidly mixed and reacted to form the product that is discharged to the surrounding liquid inside the casing 3. Thereafter the product solution is pressed (PRESSURE) through the aperture 3c thus dosing the solution to the specific purpose of use de-
15 sired. Sealing ring 8 is used in the cover 3a orifice for the plunger shaft 6a and sealing ring 9 between the piston head 6c and the casing 3.

An other example of an apparatus assembly E according to the present invention is depicted by figure 2. In a spray bottle E; E2 as shown in fig. 2 the microstruc-
20 tured device 2 is assembled to the screw cap 5 of the bottle acting as the reservoir to dilute the reactant solution. The apparatus assembly comprises two sealed containers 1, microstructured device 2, casing 3 (the bottle body), spraying device 4 and the bottle screw cap 5. The microstructured device 2 can be fitted inside the screw cap 5 of the bottle comprising an outer thread 5a and an inner thread 5b. The spraying device 4 comprises a screw cap 4a which is connected to a suction
25 hose 4b. By means of the suction hose 4b spraying device 4 is used as the solution transporting device 20; 20a. On the other hand, by the means of a screw cap 4a the spraying device is used in a puncturizing device 20; 20b of reaction launching means 20. On the top of each container 1 there is a piston 7 comprising a plunger shaft 7a and a piston head 7c. Under each container there is a prong 7d
30 that punctures diaphragm 1a when piston plunger shaft 7a is pressed. Piston 7 and prong 7d belongs both to the puncturizing device 20; 20b of reaction launching means.

When starting the operation, the bottle screw cap 5 is arranged partially inside the bottle neck. The microstructured device 2 is mounted inside the bottle screw cap 5
35 by fastening with inner thread 5b of the bottle screw cap 5. Thereafter, the screw cap 4a of the spraying device 4, is connected loosely outside the bottle screw

- cap 5 by contacting with the outer thread 5a of the bottle screw cap 5. When the screw cap 4a is connected loosely to the bottle screw cap, each plunger head 7c touches corresponding diaphragm 1a of the container 1. When the connection between spraying device 4 and outer thread 5a of the bottle screw cap 5 is fastened more and more, each prong 7d pierces diaphragm 1a and the both reactant containers 1 are emptied inside the free space 3b of the casing 3 as the plunger shaft 7a presses down. Subsequently, the diluted product solution in spray bottle E; E2 is ready to be used using the spray device 4 through the suction hose 4b thus dosing the solution to the specific point of use desired.
- 10 Yet another example of an apparatus assembly according to the present invention is depicted by figure 3. The apparatus assembly E is a syringe E3 whose main parts are: two sealed reactant containers 1, a microstructured device 2, a casing 3 and a combined means 20 for transporting and launching the reaction. The combined means 20 for transporting the solution and launching the reaction includes a manual primary piston set 6, a secondary piston set 7 and springs 7e and prongs 7d. The primary piston set 6 comprises a plunger handle 6b, a plunger shaft 6a and a piston head 6c. The secondary container piston set 7 comprises a handle 7b, connected to two plunger shafts 7a which are each connected to a piston head 7c above the container 1. The primary piston set 6 is connected to the secondary piston set 7 so, that the head 6c of the primary piston set 6 is the handle 7b of the secondary piston set 7. Under each container 1 is disposed a spring 7e and a prong 7d. Since the primary piston set 6 is connected fixedly to the secondary piston set 7 both piston sets 6,7 are used as means 20 for transporting and launching the reaction (and at the same time as a puncturizing device). As mentioned above, the means 20 for launching the reaction comprises also springs 7e and prongs 7d, below the containers, which puncture diaphragms 1a when the plunger shafts 7a are pressed.
- 30 The syringe E; E3 is operated as follows. The sealed containers 1 are placed on the top of springs 7e and the piston system is mounted on the top of the containers (START POSITION). When the primary piston set 6 is pressed down (PRESSURE) the secondary piston set 7 is also forced down. When pressing the plunger shafts 7a and heads 7c of the secondary piston set 7, prongs 7d puncture diaphragms 1a, and the solution is pushed into the microstructured device 2 where the reaction takes place. The primary piston set 6 is used to push the reagents through the microstructured device and finally the product solution through the aperture 3c, thus dosing the solution to the specific purpose of use desired. Sealing
- 35

ring 10 is fitted between the primary piston head 6c and the casing 3 and sealing rings 11 are fitted between the container piston heads 7c and container 1 walls.

According to another preferred embodiment means for launching the reaction and/or entraining liquid solvent inside the casing include a hand-powered device, wherein the reactant solution is forced through the aperture by back-pressure built up by using a hand pump pushing air with manual strokes into a space within the partially liquid-filled casing. Preferably, the casing is otherwise air-tight, but has one valve to let the incoming air in from the pump. As more air is pumped inside the casing, the air in the casing is compressed, increasing in pressure; the water is also pressurized by the now compressed air. The back-pressure may be released by opening of the nozzle valve. The pressurized liquid is then pushed out through the aperture as the air attempts to re-equilibrate with atmospheric pressure.

According to yet another embodiment means for launching the reaction and/or entraining liquid solvent inside the casing include an air-powered, pneumatic device wherein compressed air is directed inside the casing, the air pressure serving to force the fluid applied prior to operation for sealing the nozzle through the aperture.

The invention further discloses a method for preparation of the active chemical product using the above described dosing apparatus as depicted in claim 13.

The method includes the use of this ready-to-use dosing apparatus assembly as described above. The apparatus assembly including the reactants may be stored as described in the regular storage instructions for the specific reactants inside the reactant containers depending on the known chemical properties thereof.

When the desired active chemical product, preferably performic acid solution, most preferably an aqueous solution of performic acid, is needed at the point-of-use, the reactants are discharged from the reagent containers by the discharge means into the microstructured device. Preferably, discharging takes place in less than 15 seconds initiating the reaction. Thorough mixing is enabled by microchannels of the microstructured device and initiation of the reaction takes also place at the microstructured device. Depending on the reaction kinetics the reaction may be completed instantaneously. Alternatively, the reactant mixture is further led to the means for completing the reaction. The desired chemical product is discharged through the outlet of the apparatus and provided to the point-of-use, preferably within a few minutes or less.

The apparatus is operated in a batch mode. By using this kind of apparatus the chemical product can be dosed totally or partly into the target, depending on the quantity needed.

- 5 In a yet more preferred embodiment the apparatus is recycled after use. It is flushed and/or stored before taking it back to the recycling, preferably to the supplier. Once received at the point of recycling the apparatus is cleaned by flushing, refilled or the reactant containers are exchanged.

The apparatus may be enclosed inside a suitable casing for transport, storage and return to e.g. the supplier for refilling.

- 10 The method and the apparatus of the invention may be used in various fields. Preferably, the chemical product, most preferably performic acid solution, obtained is used for sanitizing purposes, preferably for disinfecting, more preferably for disinfecting surfaces and apparatus at hospitals or medical care environment such as in health care centers i.e. hospitals and medical buildings in particular.
- 15 The invention is further illustrated by the following non-restricting examples.

Example 1

Performic acid is prepared at the point of use from formic acid and hydrogen peroxide using sulphuric acid as catalyst in the apparatus assembly E;E1 according to figure 1.

- 20 The assembly E; E1 consists of two sealed reactant containers 1 the first container for aqueous solution of 75% formic acid solution, 12% sulphuric acid solution 13% H₂O and the second container 1 for 50% aqueous hydrogen peroxide solution, a commercial microreactor, casing 3 encasing the reactant containers 1 and the microreactor 2, a piston 6 composed of a plunger handle 6b, a plunger shaft 6a and
- 25 a detached piston head 6c that is connected to the microreactor 2. The top walls of reactant containers 1 are also connectable to the piston head 6c. The plunger shaft 6a has a collet 6d that is to be connected to the piston head 6c via a collet nut 6e. The bottom of each reactant container 1 is made of diaphragm 1a that is to be punctured to release the liquids out from the containers. On the top of the con-
- 30 tainers 1 there are container pistons 7 with plunger shafts 7a and piston heads 7c. Under the containers 1 there are prongs 7d that puncture the diaphragms 1a when the piston head 7c is pressed. When the prongs 7d pierce the diaphragms 1a the reactant containers 1 are emptied as the piston 7 is pressed down.

When the syringe E; E1 is taken into use, the collet 6d is pressed to collet nut 6e and the piston head 6c is pulled to the direction away from aperture 3d at the bottom of the casing 3 and water is sucked to the free space 3b inside the casing 3 through this aperture 3d. When the piston shafts 6a reach the cover 3a of the casing 3 the diaphragms 1a are punctured by prongs 6d and reactants of the containers 1 enter into the microreactor 2 where the reactants are rapidly mixed and reacted to form the aqueous performic acid product solution that is subsequently discharged to the surrounding liquid inside the syringe E1 casing 3 causing slight increase in solution temperature. Thereafter, the equilibrium solution mixture, containing diluted performic acid solution is pressed through the aperture 3d thus dosing the resulting solution to the point of use.

The prepared solution is used for disinfecting medical apparatus and cleaning facilities. As the formed solution is unstable it should be used within a few hours.

Example 2

The apparatus assembly of figure 3 is used for preparing de-icing chemical, potassium formate solution.

The apparatus assembly E comprises a syringe E; E1 with two sealed containers 378 ml of 85% by weight formic acid, 622 ml of 50% by weight potassium hydroxide, a microstructured device 2, a casing 3 of the syringe E1 and a container piston device 7. The syringe E; E1 consists of a primary piston set 6 with plunger handle 6b, plunger shaft 6a and a piston head 6c. Above the containers 1 there is a secondary container piston set 7 with container plunger shafts 7a and container piston heads 7c, wherein each piston head 7c is disposed close by the container 1 top wall. Under each container 1 there is prong 7d and spring 7e and the prong 7d can puncture a diaphragm 1a of the container 1 when the piston head 7c is pressed by the plunger shaft 7a.

When starting the operation, the sealed containers 1 are placed on the top of springs and the piston system 6,7 is mounted on the top of the containers as described above. When the primary piston set 6 is pressed down the secondary piston set 7 is also forced down. When pressing the plunger shafts 7a of the secondary piston set 7, prongs 7d puncture diaphragms 1a, and the solution is pushed into the microstructured device 2 where the reactants are rapidly mixed and reacted to form potassium formate. The primary piston set 6 is used to push the reagents through the microstructured device 2 and finally the product solution with

1000 ml of Potassium formate 50 wt % solution, through the aperture 3d, thus dosing the solution to the specific purpose of use desired.

The formed 50 wt % potassium formate product solution is used for de-icing exterior surfaces like de-icing frozen vehicle and ship door locks and vehicle wind
5 screens. The freezing point of the produced potassium formate solution is close to -60°C. Due to high exothermic heat of reaction the local warming at the point of use can be several tens of degrees.

By having reactants in room temperature, the increase of adiabatic temperature in the reaction is 158°C meaning that the solution is going to be partly evaporated
10 without cooling. On the other hand, by keeping the formate solution in the room temperature, reaction enthalpy (+ 574 kJ) of neutralisation can increase the temperature of 4.5 litres of cooling water from 20°C to 50°C. It is also possible to utilise heat formation as a separate "heat pack" and potassium formate solution for de-icing or its other applications.

15 **Example 3**

Performic acid is prepared at the point of use from formic acid and hydrogen peroxide using sulphuric acid as catalyst in the apparatus according to figure 1 with the exception of having three reagent containers instead of two.

The apparatus E; E1 consists of three sealed reactant containers one for each re-
20 actant, first for 90% aqueous formic acid solution, second for 50% aqueous hydrogen peroxide solution and third for 10% aqueous sulphuric acid solution, a commercial microreactor and otherwise the apparatus and its operation being the same as defined in example 1. The prepared solution can be used for disinfecting medical apparatus and cleaning facilities.

25 **Example batch operated apparatus assembly and their use have been described above. The following clauses setup some of the combinations of features envisaged by the present disclosure.**

1. A batch operated dosing apparatus assembly for producing an active chemical product wherein said apparatus comprises

30 v. at least two sealed reactant containers for providing at least two liquid reactants for a chemical reaction taking place inside said dosing apparatus assembly and resulting in said active chemical product,

- vi. at least one microstructured device providing a reaction space for said chemical reaction with efficient mixing for said reactants, whereby the inlet of at least one device is adapted to receive the liquid reactants from said containers (1),
 - 5 vii. a casing functionally connected to said sealed reactant containers (1) and said microstructured device(s), and optionally being capable of enclosing liquid solvent which is able to dissolve or dilute said active chemical product and
 - 10 viii. means for launching said reaction and/or carrying said liquid solvent inside said casing.
2. The dosing apparatus assembly according to clause 1 **characterized** in that means (20) for launching said reaction and/or carrying said liquid solvent include a device (7) for breaking the containers (1) partly or completely.
3. The dosing apparatus assembly according to clause 1 or 2 **characterized** in that said chemical reaction is selected from an oxidation reaction, reduction reaction, neutralisation reaction, peroxygen reaction and catalytic reaction.
- 15 4. The dosing apparatus assembly according to any of the clauses 1–3 **characterized** in that said microstructured device (2) is selected from the group consisting of a micromixer, a microreactor or a microchannel system.
- 20 5. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that the free space (3b) inside said casing (3) is filled with solvent.
6. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that said sealed reactant container (1) is provided with puncturable seal (1a).
- 25 7. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that said apparatus assembly is readily portable.
8. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that said dosing apparatus assembly is in a form of a syringe, spray bottle or a grease pump.
- 30

9. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that said active chemical product is an aqueous solution, preferably an aqueous solution product of a neutralisation or a peroxy reaction, more preferably peroxygen compound, most preferably peracid, such as performic acid.
- 5 10. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that said active chemical product is potassium formate.
11. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that said microstructured device (2) comprises channels with a hydraulic diameter of from 10 μm to 2 mm, preferably from 100 μm to 1 mm.
- 10 12. The dosing apparatus assembly according to any one of the previous clauses **characterized** in that the batch capacity of said apparatus is 50–3000 ml, preferably 100–2000 ml, more preferably 100–1000 ml, most preferably 100–500 ml.
13. A method for preparation of an active chemical product using the dosing apparatus assembly according to any of the clauses 1–12.
- 15 14. The method according to clause 13 **characterized** in that the reactants are discharged from the reactant container(s) (1) into the microstructured device(s) (2) for through mixing and reacting, and discharged further inside the casing wherefrom the active chemical product is led to the point-of-use.
- 20 15. Use of the active chemical substance made by the method of any one of the clauses 12–14 for sanitary and hygienic purposes, preferably for disinfecting, more preferably for disinfecting surfaces and apparatus at hospitals or medical care environment.

Various example embodiments have been described herein for dosing apparatus assembly, the method for using this apparatus and for the use of the active chemical substance made by the method. Those skilled in the art will understand, however, that changes and modifications may be made to those examples without departing from the scope and spirit of the claimed invention.

25

Reference numbers

	E	Apparatus assembly (generally)
	E1	Apparatus assembly (multipiston syringe)
	E2	Apparatus assembly (spray bottle)
5	E3	Apparatus assembly (syringe)
	1	container
	1a	seal of a container
	2	microstructured device
	3	casing
10	3a	top wall of a casing
	3b	space inside the casing
	3d	bottom aperture of a casing
	4	spraying device
	4a	screw cap
15	4b	suction hose
	5	screw cap (of the bottle)
	5a	outer thread
	5b	inner thread
	6	primary piston (set)
20	6a	plunger shaft
	6b	plunger handle
	6c	piston head
	6d	collet
	6e	collet nut
25	7	secondary piston (set), container piston
	7a	plunger shaft
	7b	plunger handle
	7c	piston head
	7d	prong
30	7e	spring
	8	sealing ring
	9	sealing ring

	10	sealing ring
	11	sealing ring
	20	means for launching and/or carrying liquid
	20;20a	transporting device
5	20;20b	puncturizing device

Claims

1. A batch operated dosing apparatus assembly for producing an active chemical product **characterized** in that said apparatus comprises
- 5 ix. at least two sealed reactant containers (1) for providing at least two liquid reactants for a chemical reaction taking place inside said dosing apparatus assembly (E) and resulting in said active chemical product,
- 10 x. at least one microstructured device (2) providing a reaction space for said chemical reaction with efficient mixing for said reactants, whereby the inlet of at least one device is adapted to receive the liquid reactants from said containers (1),
- 15 xi. a casing (3) functionally connected to said sealed reactant containers (1) and said microstructured device(s) (2), and optionally being capable of enclosing liquid solvent which is able to dissolve or dilute said active chemical product and
- xii. means (20) for launching said reaction and carrying said liquid solvent inside said casing (3), which means (20) include a device (7) for breaking the containers (1) partly or completely.
- 20 2. The dosing apparatus assembly according to claim 1 **characterized** in that said chemical reaction is selected from an oxidation reaction, reduction reaction, neutralisation reaction, peroxygen reaction and catalytic reaction.
3. The dosing apparatus assembly according to claim 1 or 2 **characterized** in that said microstructured device (2) is selected from the group consisting of a micromixer, a microreactor or a microchannel system.
- 25 4. The dosing apparatus assembly according to any one of the previous claims **characterized** in that the free space (3b) inside said casing (3) is substantially filled with liquid solvent.
5. The dosing apparatus assembly according to any one of the previous claims **characterized** in that said sealed reactant container (1) is provided with
- 30 puncturable seal (1a).

6. The dosing apparatus assembly according to any one of the previous claims **characterized** in that said apparatus assembly is readily portable.
7. The dosing apparatus assembly according to any one of the previous claims **characterized** in that said dosing apparatus assembly is in a form of a syringe, spray bottle or a grease pump.
8. The dosing apparatus assembly according to any one of the previous claims **characterized** in that said active chemical product is an aqueous solution, preferably an aqueous solution product of a neutralisation or a peroxy reaction, more preferably peroxygen compound, most preferably peracid, such as performic acid.
9. The dosing apparatus assembly according to any one of the previous claims **characterized** in that said active chemical product is potassium formate.
10. The dosing apparatus assembly according to any one of the previous claims **characterized** in that said microstructured device (2) comprises channels with a hydraulic diameter of from 10 μm to 2 mm, preferably from 100 μm to 1 mm.
11. The dosing apparatus assembly according to any one of the previous claims **characterized** in that the batch capacity of said apparatus is 50–3000 ml, preferably 100–2000 ml, more preferably 100–1000 ml, most preferably 100–500 ml.
12. A method for preparation of an active chemical product using a batch operated dosing apparatus assembly comprising
- at least two sealed reactant containers (1),
 - at least one microstructured device (2) which provide a reaction space for a chemical reaction and with efficient mixing for reactants to be mixed therein, whereby the inlet of at least one device is adapted to receive the liquid reactants from said containers (1),
 - a casing (3) functionally connected to said sealed reactant containers (1) and said microstructured device(s) (2), and optionally being capable of enclosing liquid solvent and

-means (20) for launching said reaction and/or carrying said liquid solvent inside said casing (3), which means (20) include a device (7) for breaking the containers (1) partly or completely, which method comprises at least the following steps:

-taking liquid solvent inside the casing (3) by means (20),

5 -breaking the containers (1) partly or completely for freeing the reactants,

-mixing the reactants in the microstructured device (2) for producing an active chemical product.

13. The method according to claim 12 which further comprises dissolving or diluting the active chemical product into liquid solvent for achieving a product solution and discarding said product solution from the dosing apparatus assembly by
10 means of means (20) such as a piston (7).

14. The method according to claim 12 or 13 wherein the reactants are discharged from the reactant container(s) (1) into the microstructured device(s) (2) for through mixing and reacting, and discharged further inside the casing wherefrom
15 the active chemical product is led to the point-of-use.

15. Use of the active chemical substance made by the method of any one of the claims 12–13 for sanitary and hygienic purposes, preferably for disinfecting, more preferably for disinfecting surfaces and apparatus at hospitals or medical care environment.

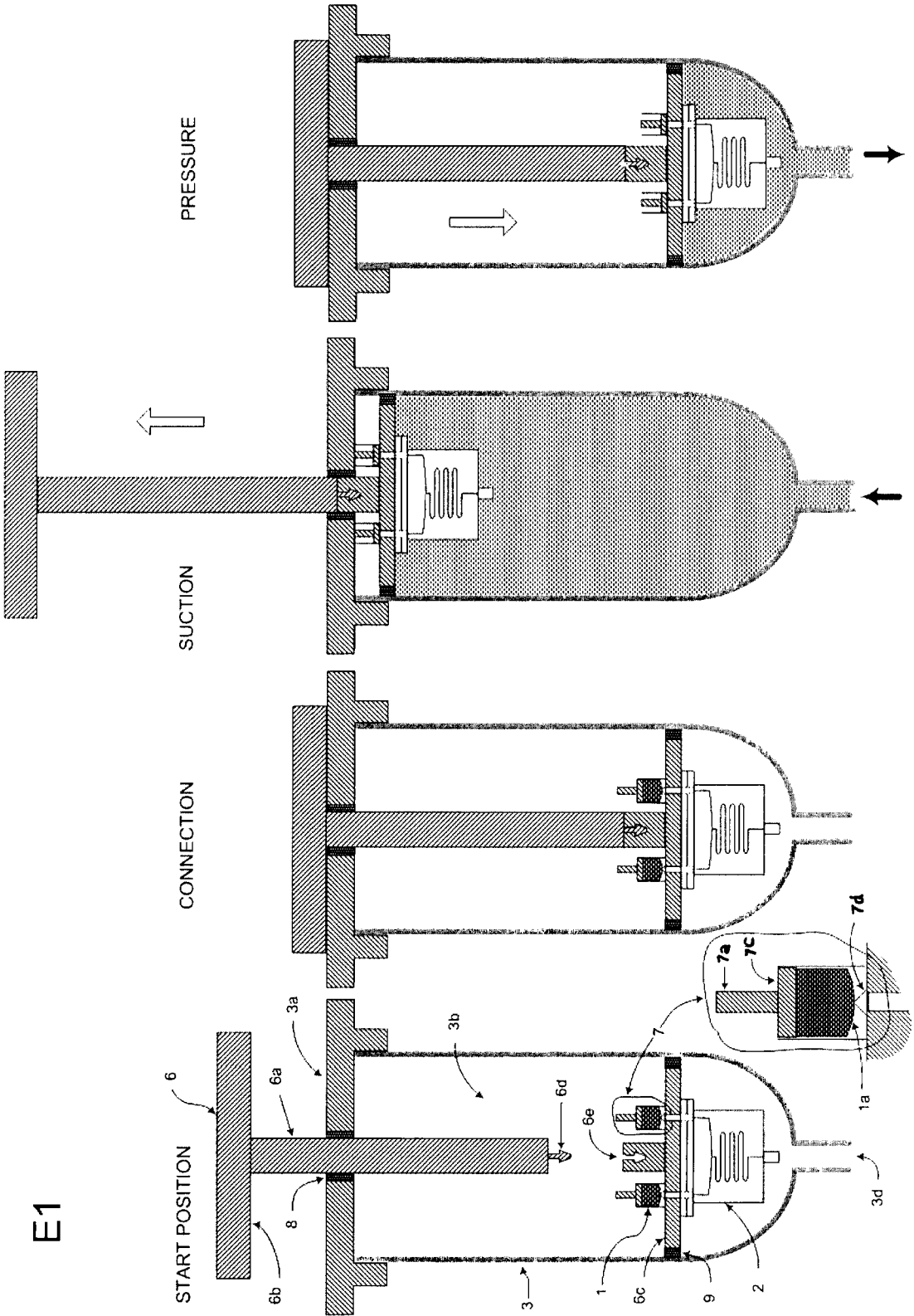


Figure 1

E2

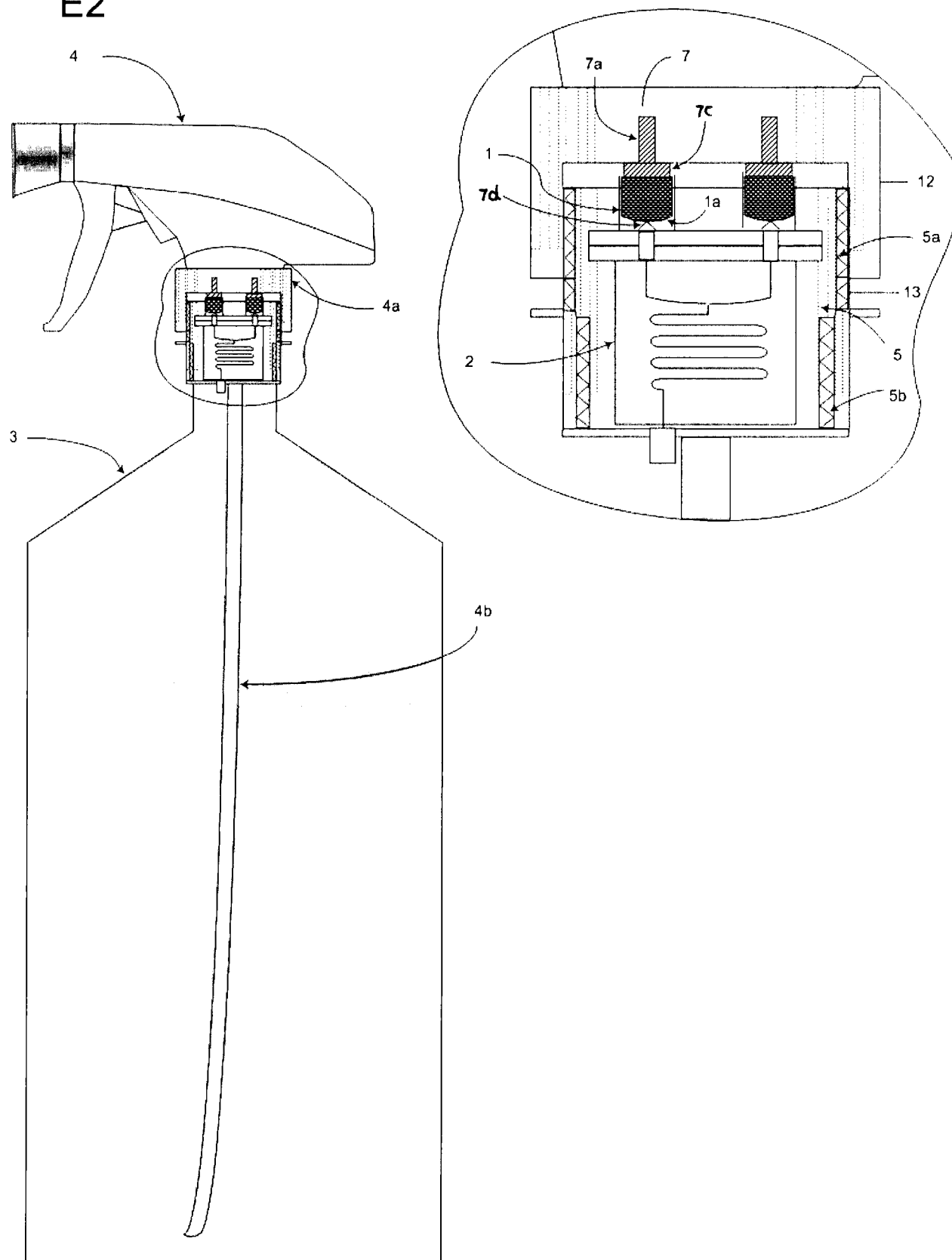


Figure 2

E3

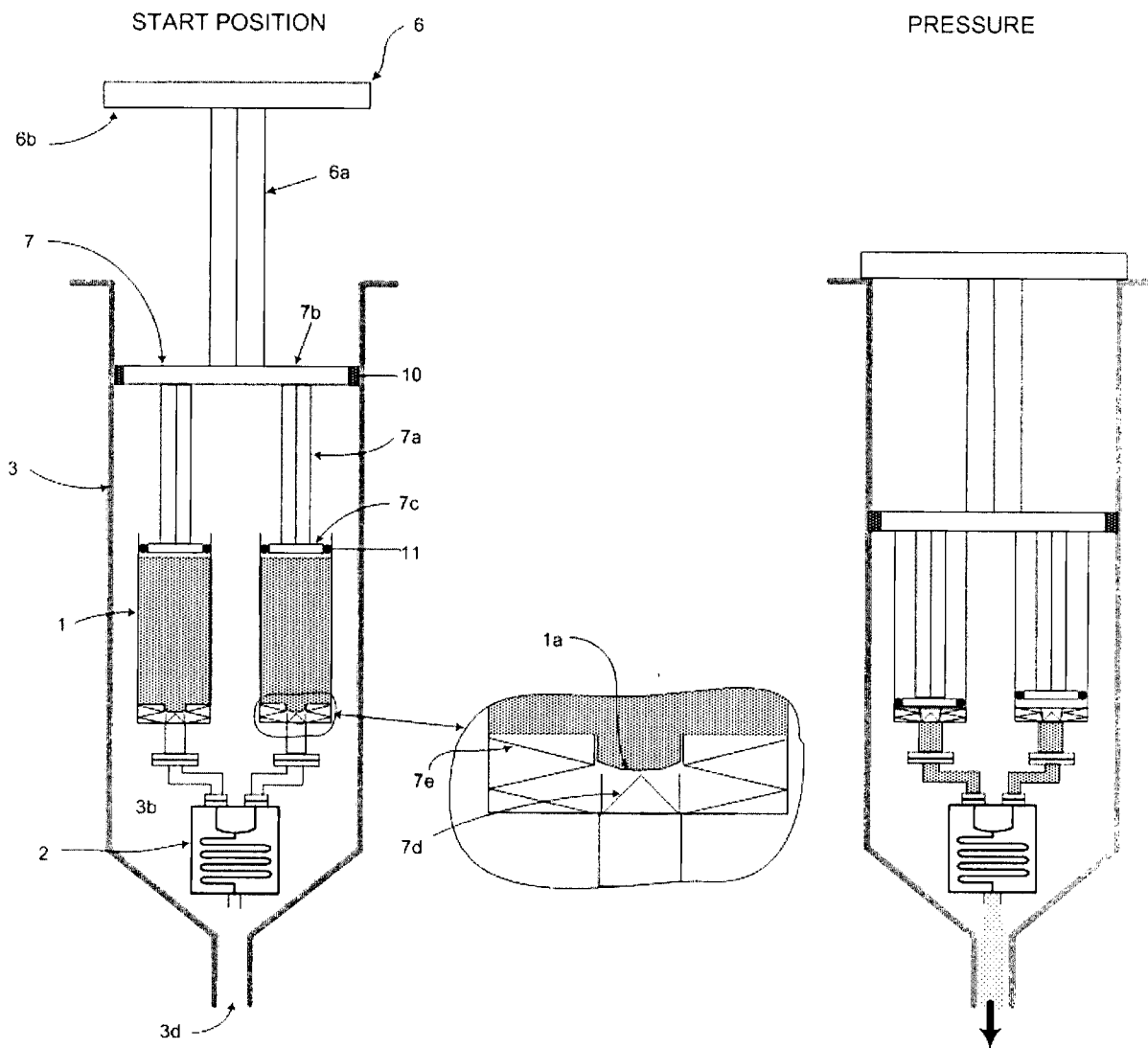


Figure 3