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(54) **Title:** MEDICAL DEVICE AND LUBRICANT COATING THEREFOR

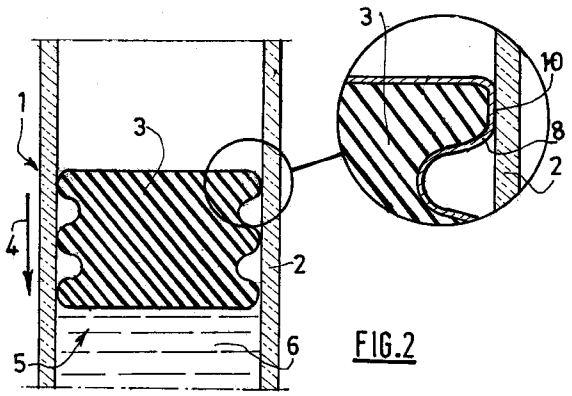
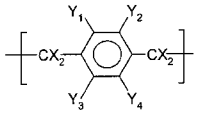


FIG.2



(I)

(57) **Abstract:** The invention relates to a medical device (1) comprising a first part (2; 3) and a second part (3; 2), movable relative to each other, a coating (8) having one contact region (10) with one of said first and second parts (2; 3), and designed to encourage the relative sliding of said first and second parts (2, 3) one relative to the other and tightness in the contact region (10), the coating (8) consisting of one polymer material comprising polymer chains having the following repeat unit, Formula (I): in which X represents a halogen, for example F, or a hydrogen, and in which Y_1, Y_2, Y_3, Y_4 each independently represent a halogen, for example Cl, or a hydrogen, characterized in that the mean thickness of said coating (8) ranges from 3 to 10 μm.

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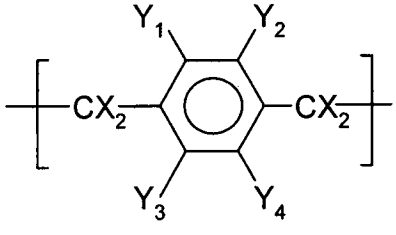
Medical device and lubricant coating therefor

The present invention relates in general to a medical device with a lubricant coating, for example a syringe, comprising two parts, for example a container and a piston, said parts being able to move one relative to the other, for example translationally and/or rotationally, when the medical device is operated.

Furthermore, the container is intended to accommodate a medical product in the liquid, gaseous, fluid, pasty or lyophilized phase, which may have a variable viscosity and is therefore able to flow, particularly because of the pressure exerted as a result of the movement of the piston relative to the container. The piston is preferably made at least partially from a viscoelastic material so as to ensure tightness in the region of contact between the container and the piston. At the same time, the volume of the medical product contained in the medical device varies, for example decreases, according to the relative movement between the two parts of the medical device.

The present invention also relates to a part for a medical device, this part being intended to cooperate with a complementary part by moving relative to said complementary part when the medical device is operated, said part being provided with a coating.

In order to improve the slip between said parts, it has been proposed for the entirety of the developed surface of one of the parts to be coated with a coating consisting of at least one polymer material, whether this is a true polymer or a copolymer, comprising polymer chains including repeats of one or more chemical units:



in which X represents a halogen, for example F, or a hydrogen,
and in which Y_1, Y_2, Y_3, Y_4 each independently represent a halogen, for example Cl, or a hydrogen.

For example, the polymer material is chosen from the group consisting of poly(p-xylylene) polymers, which may or may not be substituted, and in particular, poly(p-xylylene), poly(p-meta-chloroxylylene), poly(p-ortho-chloro/meta-chloroxylylene) and poly(p-difluoroxylylene). The latter four polymer materials are manufactured and sold by UNION CARBIDE CORPORATION, or by SPECIALTY COATING SYSTEMS, under the names Parylene N, Parylene C, Parylene D and Parylene AF_4, respectively.

For information regarding the synthesis of these particular polymer materials, particularly using chemical vapour polymerization (CVP), on their various properties and on their main uses or applications, reference may usefully be made to the following documents, the respective contents of which are incorporated as required into this description: US 3,288,728, US 3,342,754, US 3,379,803, US 3,472,795, US 4,225,647, US 3,300,332 and US 6,270,872.

These polymer materials have various properties, for example imperviousness to gases, for example oxygen, and to dry-lubricating liquids, for example water, which make them particularly attractive for use in numerous biomedical applications, particularly for certain medical devices.

Unlike a conventional polymer material, a polymer material of the poly(p-xylylene) type is not employed by injection, dissolving or suspending in a solvent, but is used by depositing it onto the part by a direct dry vacuum deposition process using the following protocol:

(a) use is made of a polymerization intermediate of the polymer material, in this instance of a cyclic dimer form of the aforementioned chemical unit, in solid and divided form,

(b) the dimer is vaporized under vacuum (1 mm of mercury for example) and at approximately 150°C for example,

(c) the vaporized dimer is then pyrolized, still under vacuum but at a higher temperature, for example at 650°C, in order to obtain the reactive monomer form corresponding to the aforementioned dimer and to the aforementioned chemical unit, and

(d) the reactive monomer is deposited directly on the entire accessible developed surface of the part, both internal and external, and polymerized at ambient temperature under a low vacuum, in a method akin to the vacuum deposition of a thin metal layer, so as to obtain a continuous coating of (substituted or unsubstituted) poly(p-xylylene) of relatively uniform

thickness, completely (with no discontinuity) covering the part of the medical device.

Various equipment and corresponding operating procedures are nowadays available on the market for the purposes of obtaining a poly(p-xylylene) coating and, by way of example, reference may be made to the equipment sold by COMELEC SA, CH-2301 La Chaux de Fonds, Switzerland, or alternatively to the PDS 2010 Labcoter 2 equipment sold by SPECIALTY COATING SYSTEMS.

The coating thus obtained, made of relatively crystalline polymer, adheres to the part directly or indirectly. Because of its slip characteristics, the coating facilitates the relative movement between the two parts of the medical device. In addition, the elastic behaviour of the coating allows it in a resilient manner to accommodate the deformations and stresses imposed on the part provided with it, for example the piston, as it slides in the container. Thus, tightness in the region of contact between the piston and the container can be guaranteed to be maintained.

Adhesion between the coating and the part may be direct, particularly by means of chemical bonds formed at the time of deposition and polymerization of the reactive monomer, between said part and the polymer material, or indirect, by way of a tie layer or primer layer applied beforehand to the surface that is to be coated, if appropriate after that surface has been cleaned or prepared.

According to document US-A-2005/0 010 175, it has been proposed that the polymer material coating of the poly(p-xylylene) type has a thickness ranging from 0.25 μm to 1 μm, it being possible for a coating thickness of 0.10 to 76 μm to be obtained in a single stage.

In the experience of the Applicant, this thickness range seems inappropriate for most medical devices, particularly of the syringe type. This is because with this range of thicknesses, when the two parts of the medical device move relative to one another, the coating breaks, tears or breaks up. This permanently worsens the surface finish of the coated part, at the region of contact between the two moving parts, thus increasing resistance to movement, or friction, between said two parts.

Document US 5,000,994 devotes itself to medical devices, of the syringe type, comprising a part of the piston type consisting of a viscoelastic or elastomeric material and at least one contact region of which provides tightness

with the container of the medical device, in this instance the syringe body. More specifically, that document is concerned with the automatic fitting of pistons into syringe bodies and with the circulation or transportation for that purpose of loose pistons, in contact with one another, in gravity-fed feeders, for example those with a vibrating bowl. It is then found that, because of the elastomeric nature of the material of the pistons, on the one hand, and because of their sterilization treatment, on the other hand, these pistons are liable to rub together significantly or even clump together when they are being circulated or transported loose, thus altering and reducing the speed at which they flow or circulate loose.

For the purposes of reducing the coefficient of friction between pistons, it is proposed in US 5, 000, 994 that they be coated with a coating between 0.1 and 2.0 μm thick.

Such a thickness is undoubtedly suitable for reducing the coefficient of friction between pistons, outside the medical devices that incorporate them, in industrial processes where they are handled loose. However, as stated above, a coating as thin as this is unable to withstand the friction forces involved in the relative movement between two parts of a medical device, and is unable to generate a durable and mechanically strong seal therebetween, particularly once the medical device has been sterilized.

According to document US 5,075,174, for the purposes of altering the surface properties of diverse and various seals, made of viscoelastic or elastomeric material, particularly in terms of the attraction of particles or dust in suspension in the air, it has been proposed that these be coated with a poly(p-xylylene) coating with a thickness ranging from 0.1 to 3 μm and preferably from 0.5 to 2 μm.

That document does not mention whether the seals in question ensure tightness between two moving parts.

As stated previously, the thicknesses considered for the coating of a first part movable relative to a second part seem far too low.

According to document US 5,354,286, a plastic or metal container is provided with a polyparaxylylene coating 0.25 to 25 μm thick so as to reduce its coefficient of friction. It is clearly evident from the foregoing explanations that a coating of the order of 0.25 μm thick is unsuitable for the application of the invention. Furthermore, a 25 μm coating would be too thick, making it more rigid and adversely affecting its elastic behaviour. The result would be an

adverse effect on the desired function of tightness in the region of contact between the two moving parts. Furthermore, nothing in document US 5,354,286 is able to demonstrate that a narrower and better targeted range of values would provide an answer to the problems that the invention proposes to solve.

The medical devices as previously defined and discussed therefore require substantial improvements, in respect of the following requirements, which are sometimes contradictory, as far as the coating is concerned.

The viscoelastic material of which the piston of a medical device such as syringe may be made is generally an elastomeric material which alters, in particular degrades chemically over time. This possible degradation is sometimes initiated by the processes used to sterilize the medical devices containing them, for example bringing them into contact with ionizing radiation. Such degradation alters the surface properties of the elastomeric material, for example the adhesion or friction with respect to one of the other parts of the medical device. Over time, that is to say as soon as the medical device has been filled with the medical product, and in particular when it is used or operated, it is therefore necessary for a coating to effectively isolate the region of contact between a first part of the device made of such a viscoelastic material and a second part of the device, intended to cooperate with said first part, so that the surface characteristics, including the coefficient of friction, of the region of contact between the two parts of the medical device, can be maintained over time, even after prolonged storage, regardless from the fact that the properties of said viscoelastic material may have been adversely affected over time.

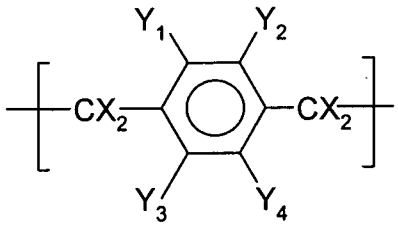
Conversely, increasing the thickness of the coating too far stiffens the latter, making it brittle, altering its viscoelastic properties and having an adverse effect on its tightness function.

It is therefore an object of the present invention to provide a thickness of coating which is appropriate to reconcile the gliding function between two complementary parts with the maintaining of tightness in the contact region of said parts, static tightness over time and dynamic tightness when using the medical device, whether the coating be provided on a first part such as a piston, on a second part such as a container or on an intermediate part located between said first and second parts.

According to the present invention, it has to these ends been found that the mean thickness of the coating needs to range from 3 to 10 μm, preferably from 3 to 5 μm.

Such a thickness also has the advantage of ensuring that, regardless of the profile, shape or surface characteristics of the coated part, at the end of the process of depositing/polymerizing the polymer material, the coating covers the entirety of the part over the desired region, namely at least the region corresponding to the contact region, with no discontinuity, and does so durably.

A first aspect of the present invention is a medical device comprising at least a first part and a second part, said first and second parts being able to move one relative to the other and determining between them at least one contact region, at least one of said first and second parts being provided with at least one coating designed to at least encourage the relative sliding of said first and second parts one relative to the other and tightness in said contact region, and said coating consisting of at least one polymer material comprising polymer chains having the following repeat unit:



in which X represents a halogen, for example F, or a hydrogen,
and in which Y_1, Y_2, Y_3, Y_4 each independently represent a halogen, for example Cl, or a hydrogen,
characterized in that the mean thickness of said coating ranges from 3 to 10 μm.

The medical device of the invention allows to have decreased activation, sustainable and final forces for moving a first part relative to a second part, for example for moving a piston within the container in which it is lodged, without having to add a lubricant and while preserving the tightness at the contact region between said two parts. For example, in a medical device such as a syringe, the piston must be able to be moved relative to the container or syringe body, through a gliding movement, while at the same time ensuring

the tightness with said container, so that all of the product to be administered escapes only via the distal end of the container and does not leak out of said container via the piston at the proximal end of the container. The medical device of the invention, thanks to a specific coating having a specific thickness range at the contact region between the piston and the container, allows the successful completion of these two relatively incompatible requirements.

In this application, the distal end of a component or of a device means the end furthest away from the hand of the user and the proximal end means the end closest to the hand of the user. Similarly, in this application, the terms "in the distal direction" and "distally" mean in the direction of the injection, and the terms "in the proximal direction" and "proximally" mean in the direction opposite to the direction of injection.

Moreover, with the medical device of the invention, it is possible to decrease the total amount of lubricant, for example silicone oil, that is necessary in such a medical device.

In consequence, the medical device of the invention allows to limit the risk of interaction between a lubricant, for example silicone oil, and the therapeutic molecules potentially stored in the container of the medical device prior to delivery to a patient.

Preferably, the mean thickness of said coating ranges from 3 to 5 μm.

In an embodiment of the invention, said first part is a container intended to accommodate a medical product and said second part is a piston movable in said container in order to vary the volume of said medical product contained in said container.

In an embodiment of the invention, said second part consists of a viscoelastic material designed to encourage tightness at said contact region.

In an embodiment, the first part is a container intended to accommodate a medical product, said medical device comprising a piston movable in said container, said second part being an intermediate part located between said container and said piston.

Said coating may be provided at least on said container.

In another embodiment of the invention, said coating is provided at least on said piston.

In a further embodiment of the invention, said coating is provided at least on said intermediate part.

Preferably, said coating is continuous and elastic.

Said polymer material is preferably chosen from the group consisting of poly(p-xylylene), poly(p-meta-chloroxylylene), poly(p-ortho-chloro/meta-chloroxylylene) and poly(p-difluoroxylylene).

In an embodiment of the invention, said contact region further includes a lubricant other than said coating.

In an embodiment of the invention, said coating provided on said container or on said piston or on said intermediate part is at least partially covered with said lubricant.

In another embodiment of the invention said piston or said container or said intermediate part, not provided with said coating, is at least partially covered with said lubricant.

In an embodiment of the invention said lubricant contains silicone.

In another embodiment of the invention, said coating is designed to have at least one outer surface relative to which at least one of said first and second parts is intended to move, said outer surface having a mean roughness Ra of less than 2.5 µm, preferably less than 2 µm and, for example, of the order of 1.0 µm.

Preferably, said polymer material consists of poly(p-meta-chloroxylene).

In an embodiment of the invention, said first part is made out of glass. For example, said first part is a glass container.

In an embodiment of the invention, the medical device includes an injection device.

The present invention is now described with reference to the attached drawings in which:

- Figure 1 depicts, schematically and in cross section, a portion of a medical device considered by the present invention and according to a first embodiment thereof,

- Figure 2 depicts, again schematically and in cross section, a portion of a medical device according to a second embodiment of the invention.

In general and with reference to Figures 1 and 2, a medical device 1 considered by the present invention, for example a syringe, comprises a first and a second parts 2 and 3, one being complementary to the other, for example a piston 3 housed in a container 2, the piston 3 and the container 2 being in contact with one another via a contact region 10. The piston 3 and the container 2 are able to move one with respect to the other in a predetermined gliding movement 4, for example translationally and/or rotationally. The

container 2 is intended to accommodate a medical product 6 in the liquid, gaseous or fluid phase, the volume of said product 6 varying according to the movement of the piston 3 with respect to the container 2. In particular, for administering the product 6, the piston 3 is caused to move distally along arrow 4 of figure 1 in order to push the product 6 out of the container 2. The piston 3 is designed to deform in order to tighten the contact region 10. For example on figure 2, at least part of the developed surface of the piston 3, which corresponds to the contact region 10, is provided with a coating 8 which is continuous, intrinsically elastic and firmly secured to the piston 3.

According to Figure 1, in a first embodiment, the piston 3 comprises an independent seal 9 housed in a groove 11 made in the piston 3, which is made of viscoelastic material, for example of elastomer, encouraging deformation of the piston 3 and therefore tightening the contact region 10. The seal 9 is also made out of a viscoelastic material, for example an elastomer, in order to ensure tightness at the contact region 10. With reference to Figure 1, the seal 9 is provided with a coating 8.

According to Figure 2, in a second embodiment, the piston 3 is made in its entirety of a viscoelastic material, for example an elastomer.

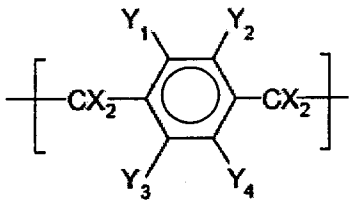
Irrespective of the embodiment considered, the contact region 10 between the container 2 and the piston 3 also determines a region of gliding contact between the piston 3 and the container 2.

According to the invention, and with reference to Figure 2, the container 2 and the piston 3 determine a contact region 10 which is provided with a coating 8. On the example shown on this figure, the coating 8 is provided on the piston 3. According to another embodiment which has not been depicted, the coating 8 is provided on the container 2. According to other alternative forms of embodiment which have not been depicted, the coating 8 may be formed of two individual coatings, one provided on the container 2 and one on the piston 3. According to another embodiment which has not been depicted, the coating is provided on one or on the two faces of an intermediate part located between the piston and the container.

The coating 8 of the medical device 1 of the invention encourages the gliding of the piston 3 relative to the container 2 at the time of administration of the product 6. Moreover, the coating 8 also ensures static and dynamic tightness at the contact region 10 of the two complementary parts, namely the piston 3 and the container 2. In particular, before use of the medical device 1,

for example during storage, the coating 8 ensures the static tightness between the piston 3 and the container 2 by preventing the leakage of the product 6 at the contact region 10 between the piston 3 and the container 2. When the medical device 1 is in use, the coating 8 ensures the dynamic tightness between the piston 3 and the container 2 by preventing the leakage of the product 6 at the contact region 10 between the piston 3 and the container 2 while the piston 3 is moving relative to the container 2.

According to the invention, the coating 8 consists of at least one polymer material comprising polymer chains consisting of the following repeat unit:



in which X represents a halogen, for example F, or a hydrogen, and in which Y_1, Y_2, Y_3 and Y_4 each independently represent a halogen, for example Cl, or a hydrogen. This coating 8 according to the invention is obtained by dry vacuum deposition/polymerization at ambient temperature, as described above.

The coating 8 according to the invention has a thickness ranging from 3 to 10 μm. Hence, when the contact region 10 is provided with two individual coatings 8, one provided on the container 2 and one on the piston 3, the thickness of the coating 8 of the medical device will therefore be the sum of the thicknesses of each individual coating 8.

Starting with the appropriate dimer, and using equipment as identified hereinabove, the person skilled in the art will know how to deposit and control a predetermined thickness of the polymer material adopted, particularly by varying the time for which the part that is to be coated is exposed to the reactive monomer form of the poly(p-xylylene) chosen. Furthermore, a person skilled in the art knows that the rate of deposition/ polymerization is directly proportional to the square of the reactive monomer concentration, and inversely proportional to the absolute temperature of the part exposed to the monomer, this information allowing him to modify and control the thickness of the coating deposited on the part.

The present invention considers various substrates or viscoelastic materials to be appropriate to the deposition of a coating 8 as previously defined, these being various natural or synthetic elastomers: silicones, nitrile-based elastomers, natural or synthetic rubber, fluorocarbon elastomers, polyurethanes. As a preference, the invention will devote itself to bromobutyl and chlorobutyl synthetic elastomers.

By way of example, the mean thickness of the coating 8 ranges from 3 to 10 μm and preferably from 3 to 5 μm.

Such a specific thickness range allows a smooth gliding of two complementary parts relative to each other while ensuring tightness at the contact region between said two complementary parts.

As stated above, the polymer material is preferably chosen from the group consisting of poly(p-xylylene), poly(p-meta-chloroxylylene), poly(p-ortho-chloro/meta-chloroxylylene) and poly(p-difluoroxylylene). As a preference, the polymer material consists of poly(p-meta-chloroxylylene).

By implementing the invention, it is possible, to a significant extent, to limit or even eliminate the amount of lubricant other than the aforementioned polymer material, for example silicone oil, customarily used at the contact region 10 of sliding contact between the piston 3 and the container 2.

According to the present invention, it has also been discovered that the roughness, and therefore the surface finish, between the container 2 and the piston 3 of the medical device 1 in the contact region 10 of sliding contact with the other part 2, is important in giving the coating 8 the desired performance and function, and this, independently of the thickness of the coating 8, provided said thickness ranges from 3 to 10 μm as defined in the present invention. According to an embodiment of the invention, the outer surface finish of the coating 8 has a mean roughness R_a of less than 2.5 μm and preferably less than 2 μm and, more preferably still, less than 1.5 μm, for example of the order of 1.0 μm.

In the present application, the roughness is measured according the following method : roughness measurements done in triplicate are performed by using a profiler Wyko NT 1100 (Veeco Instruments Inc. Tucson USA) on scans 370 μm x 240 μm with a VSI mode (Vertical Scanning Interferometry). The calibration of the apparatus is performed following the procedure WI 7.6-20 using measuring instruments traceable to the National Institute of Standards and Technology (NIST).

A roughness of less than 2.5 µm, measured as described hereinabove, for the coating 8 of a medical device of the invention allows a smooth gliding of a such coated part, like a piston, relative to a complementary part, like a container.

The present invention will now be illustrated with the following examples.

Example 1 :

The following test protocol is performed on a medical device 1 of the syringe type, according to the second embodiment depicted in Figure 2 of the present application.

The container 2 is a glass syringe body accommodating a piston 3 able to move translationally 4 inside the container 2. The piston 3 is made of a viscoelastic material such as bromobutyl rubber by West Company, or chlorobutyl rubber by West Company.

Various medical devices, or container-piston systems, were tested: some with non coated pistons, others with coated pistons. The coated pistons 3 were coated with a coating 8 as previously defined, in which the polymer material is poly(p-meta-chloroxylylene) (Parylene C). Regarding the coated pistons, several thicknesses of coating 8, and several surface finishes or roughnesses of the exterior surface of this coating 8 were tested, as summarized in Table 1 below.

Table 1 : configurations of pistons A, B1, B2, B3 and C

Piston reference	Viscoelastic substrate	Coating	Coating thickness	Surface finish
A (comparative)	Bromobutyl rubber	No	---	Smooth Ra = 0.7 µm Rt = 11.4 µm
B1 (invention)	Bromobutyl rubber	Yes	3 µm	Smooth Ra = 0.9 µm Rt =12.0 µm
B2 (invention)	Bromobutyl rubber	Yes	3 µm	Rough Ra = 3.1 µm Rt = 24.0 µm
B3 (comparative)	Bromobutyl rubber	Yes	0.5 µm	Smooth Ra = 1.8 µm Rt= 18.7 µm
C (comparative)	Chlorobutyl rubber	No	---	Smooth Ra = 0.7 µm Rt = 11.0 µm

The surface finishes of the coatings 8 of the coated pistons 3 were examined by enlarging them using a scanning electron microscope, observed on a scale of 10 to 20 µm.

The roughness measurements were made using a profiler Wyko NT 1100 (Veeco Instruments Inc. Tucson USA) over an analysis area measuring 370 µm x 240 µm with a VSI mode. Ra represents the mean roughness (the arithmetic mean of the various values of a roughness profile) and is expressed in µm. Rt represents the maximum peak-to valley height in a roughness profile and is expressed in µm.

Tests (Activation Gliding Force tests) were performed to determine the necessary forces for moving each piston 3 with respect to the container 2 in which it is housed. These tests were performed using a LLOYD-CB190 tensile testing machine dynamometer using NEXYGEN operating software, according to two test protocols outlined briefly below.

Activation Gliding Force (AGF) tests were applied on containers 2 filled with 1 mL of demineralised water and each plugged with one piston 3 to be tested (coated or uncoated). Each container 2-piston 3 system was tested 32 times in order to ensure the reproducibility and to validate the results. To prepare the 32 syringes for a system, and particularly to insert the pistons 3 in the containers 2, a Gröninger machine was used.

These gliding tests made it possible to establish the value of various friction forces referenced B, S and F, respectively:

- the friction force B is the force required, under static conditions, to break the contact at the contact region 10 between the piston 3 and the container 2,

- the friction force S is the force required, under dynamic conditions, for moving the piston 3 in the container 2. The friction force S is measured half way of the piston travel. In order to measure the friction force S, the container 2 was used filled with water,

- and the friction force F is the force required, again in dynamic mode, to move the piston 3 when it reaches the end of its travel in the container 2. Just as when measuring the friction force S, the friction force F is measured with a container 2 empty of medical product 6 but initially filled with water.

In order to study the evolution of the interface, namely the contact region 10, between the piston 3 and the container 2, samples undergo an accelerated aging in a climatic room. The conditions of the Heraeus climatic room were a temperature of 40°C and a humidity rate of 75%. The systems under assessment were placed in the climatic room during 1, 3 and 6 months.

The results obtained were as follows:

1. Piston surface finish

The surface state of the pistons was observed before any functional test and before any ageing.

It was observed that piston A, which had no coating according to the invention, had a relatively rough surface finish with peaks and troughs. The coatings 8 of the invention on pistons B1 and B2 had relatively smooth and

uniform surface finishes, and the coating on the piston B3 had a relatively rough surface finish with various irregularities.

During the CVP deposition/polymerization process, it was possible to control the surface finish of the coating obtained by intermingling the pistons 3 with one another during the CVP process, on the one hand, and by intermingling them with inserted elements (inert parts) mixed in with the pistons 3 while they were being intermingled.

2. Gliding test

Pistons B1 and B2 with their coatings 8 according to the invention were fitted and assembled in a glass container 2, such as a syringe body, coated on its internal surface with a layer of silicone at a rate of 4 μg per cm^2 ± 1. No lubricant of the silicone type was added to the pistons B1 and B2. The syringes 2 thus assembled were placed in an ageing chamber for one month at 40°C with a relative humidity RH of 75%.

The friction forces B, S and F were measured by the protocol described before, using the aforementioned equipment, at a rate of travel of 380 mm/min. Each measurement of the friction force B, S and F was repeated 30 times. The results obtained are collected in Table 2 below. The bracketed values correspond to the standard deviation.

Table 2 : Activation Gliding Forces, Pistons B1 and B2, 1 month ageing

Silicone/internal surface of syringe		4 μg/cm²	4 μg/cm²	4 μg/cm²
Force (N)		B	S	F
Piston	B1	3.0 (0.4)	3.4 (0.5)	2.8 (0.6)
	B2	2.6 (0.3)	4.3 (0.8)	4.3 (0.8)

The results obtained show that, for the same thickness of coating 8 according to the invention, namely 3 μm, the friction forces S and F are lower for piston B1 which is said to have a "smooth" surface finish of its coating 8 and a relatively low roughness value (Ra = 0.9 μm, see Table 1) than they are for piston B2, the surface finish of the coating 8 of which is said to be "rough" and

which has a roughness value higher (Ra = 3.1 µm, see Table 1) than that of the piston B1.

In conclusion, it is possible to affirm that, for the same thickness, a coating 8 with a relatively smooth surface finish is preferable for limiting friction and optimizing the sliding of the piston 3 in the container 2.

Example 2

The same gliding tests as in Example 1 were repeated using pistons B1 and B3. The results obtained are recorded in Table 3 below, T = 0 meaning that the measurement was taken prior to ageing, and T = 1 meaning that the measurement was taken after one month of ageing at 40°C with a relative humidity RH of 75%.

Table 3 : Activation Gliding Forces, pistons B1 and B3, 1 month ageing

Silicone/internal surface of syringe		4 µg/cm²	4 µg/cm²	4 µg/cm²
Force (N)		B	S	F
Piston	$B1_{T=0}$	2.1 (0.1)	2.5 (0.3)	2.6 (0.3)
	$B1_{T=1}$	3.0 (0.4)	3.4 (0.5)	2.8 (0.6)
	$B3_{T=0}$	2.5 (0.5)	3.8 (1.0)	3.0 (1.0)
	$B3_{T=1}$	2.9 (0.4)	4.1 (0.4)	2.9 (0.3)

At the end of the pistons' 3 travels, it was found that the coating 8 of the invention on piston B1 had maintained its continuity, whereas the coating on piston B3 had broken.

Significantly, after one month of ageing, the friction force S was lower for piston B1 than for piston B3.

The friction forces B, S and F measured on piston B3 varied far more than with piston B1, with the two pistons 3 covering the same travel, this being because of the local breakage of the coating of piston B3, giving rise to unpredictable sliding in the region 10 of contact between the piston B3 and the syringe body 2.

Example 3

In this example, tests are carried out in order to determine whether addition of various amounts of a silicone lubricant has an influence on the medical device 1 of the invention, in the case where said silicone lubricant is added both on the pistons 3 and on the internal surface of the containers 2.

The pistons A, B1, B2 and C of example 1 were coated by spraying respectively various quantities, respectively 5 $\mu g/cm^2$, 15 $\mu g/cm^2$ and 50 $\mu g/cm^2$, of a silicone-based lubricant (with a viscosity of 1000 cst) and were assembled in glass containers 2 themselves coated on their internal surface with a 50 $\mu g/cm^2$ coating of silicone.

The silicone amount was measured prior to any AGF test. This measurement was done in order to quantify the silicone amount in the system i.e. silicone on the piston 3 and silicone on the internal surface of the container 2, and thus, to define the low limit for the silicone amount acceptable for functional testing (AGF test).

The same experimental protocol as that defined hereinabove was used, with three different lengths of ageing period, namely:

- one month in an ageing chamber at 40°C with a relative humidity RH of 75% for pistons A, B1 and C according to Table 4,
- three months in an ageing chamber at 40°C with a relative humidity RH of 75% for pistons A, B2 and C according to Table 5, and
- six months in an ageing chamber at 40°C with a relative humidity RH of 75% for pistons A and B2 according to Table 6.
-

The results are collected in the following tables 4-6.

Table 4 : Activation Gliding Forces, pistons A, B1, C, 1 month ageing

Silicone/internal surface of container		50 µg/cm²			50 µg/cm²			50 µg/cm²		
Silicone/piston		5 µg/cm²			15 µg/cm²			50 µg/cm²		
Force (N)		B	S	F	B	S	F	B	S	F
Piston	$A_{T=0}$	6.1 (0.6)	1.1 (0.5)	7.0 (3.1)	6.0 (0.8)	0.9 (0.3)	6.9 (3.0)	6.0 (0.7)	0.8 (0.2)	6.5 (2.7)
	$A_{T=1}$	9.1 (0.6)	2.0 (1.0)	5.0 (2.1)	9.3 (2.1)	2.8 (1.0)	4.5 (1.3)	9.0 (0.5)	2.2 (1.0)	4.1 (1.6)
	$B1_{T=0}$	2.1 (0.1)	1.2 (0.3)	2.5 (1.1)	2.1 (0.1)	1.2 (0.3)	2.1 (1.2)	2.0 (0.1)	1.2 (0.3)	2.1 (1.0)
	$B1_{T=1}$	2.3 (0.2)	1.2 (0.3)	2.3 (0.5)	2.3 (0.1)	1.2 (0.2)	1.2 (0.4)	2.3 (0.2)	1.2 (0.1)	1.9 (0.4)
	$C_{T=0}$	3.8 (0.5)	0.8 (0.3)	2.5 (1.1)	3.6 (0.5)	0.8 (0.4)	4.2 (2.2)	3.6 (0.4)	0.8 (0.3)	3.3 (1.8)
	$C_{T=1}$	5.0 (0.9)	1.0 (0.3)	3.2 (1.5)	4.7 (0.7)	1.1 (0.3)	3.5 (1.4)	4.8 (0.8)	1.0 (0.2)	2.7 (1.2)

Table 5 : Activation Gliding Forces, Pistons A, B2 and C, 3 months ageing

Silicone/internal surface of container		50 µg/cm²			50 µg/cm²			50 µg/cm²		
Silicone/piston		5 µg/cm²			15µg/cm²			50 µg/cm²		
Force (N)		B	S	F	B	S	F	B	S	F
Piston	$A_{T=3}$	9.3 (2.3)	1.2 (0.2)	3.5 (1.5)	12.2 (6.8)	1.3 (0.3)	3.1 (1.3)	10.2 (5.1)	1.2 (0.4)	2.8 (0.9)
	$B2_{T=3}$	2.3 (0.3)	1.5 (0.7)	1.8 (0.4)	2.2 (0.2)	1.5 (0.1)	1.9 (0.5)	2.2 (0.2)	1.4 (0.2)	1.9 (0.4)
	$C_{T=3}$	5.9 (0.3)	1.0 (0.3)	1.6 (0.7)	6.1 (1.3)	1.0 (0.7)	1.7 (0.7)	5.7 (1.3)	1.2 (0.4)	2.2 (1.4)

Table 6 : Activation Gliding Forces, Pistons A and B2, 6 months ageing

Silicone/internal surface of container		50 µg/cm²			50 µg/cm²			50 µg/cm²		
Silicone/piston		5 µg/cm²			15µg/cm²			50 µg/cm²		
Force (N)		B	S	F	B	S	F	B	S	F
Piston	$A_{T=6}$	15 (7.0)	1.8 (1)	2.7 (2)	12 (1.8)	1.1 (0.4)	1.9 (0.9)	12.1 (1.9)	1.3 (0.6)	2.2 (1.2)
	$B2_{T=6}$	2 (0.5)	1.6 (0.3)	3 (1.5)	2 (0.4)	1.6 (0.4)	2.5 (0.7)	2.1 (0.4)	1.5 (0.3)	2.8 (0.9)

According to Table 4, it can be observed that the friction forces B and F are reduced by as much as a factor of 3 for piston B1 according to the invention, compared with pistons A and C, and that this is true independently of the amount of silicone lubricant carried on the pistons. It is therefore possible, thanks to the invention, to reduce significantly the amount of silicone lubricant carried in the medical device, and to do so without adversely affecting the sliding of the piston 3 with respect to the syringe body 2 or container.

According to Table 5, the lowest friction forces B were obtained with piston B2. In consequence, after prolonged ageing, thanks to the coating 8 according to the invention, it still remains possible to reduce the amount of silicone lubricant carried on the piston 3 while at the same time maintaining or even improving the sliding of the region 10 of contact between the piston 3 and the syringe body 2 or container.

These results are even more noticeable after six months of ageing, as shown by Table 6.

Example 4

The test protocol of Example 3 was repeated with pistons A, B1, B2 and C, with various levels of lubrication thereof, these levels being expressed by weight of silicone employed. The results obtained are given in Tables 7 and 8 below.

Table 7 : Activation Gliding Forces, Pistons A, B1 and C

Silicone/internal surface of container		4 µg/cm²			4 µg/cm²			4 µcm²		
Silicone/piston		5 µg/cm²			15 µg/cm²			50 µg/cm²		
Force (N)		B	S	F	B	S	F	B	S	F
Piston	$A_{T=0}$	6.6 (0.6)	5.8 (1.0)	4.7 (1.0)	7.2 (0.5)	6.9 (1.9)	4.7 (1.9)	6.6 (0.8)	6.5 (1.5)	4.0 (1.5)
	$A_{T=1}$	12.0 (2.3)	8.4 (2.0)	3.2 (1.3)	11.0 (0.8)	8.0 (1.9)	3.3 (1.0)	10.7 (1.2)	6.7 (1.5)	3.6 (1.7)
	$B1_{T=0}$	2.2 (0.2)	2.7 (0.4)	2.7 (0.4)	2.2 (0.2)	3.0 (0.6)	3.0 (0.6)	2.1 (0.1)	2.6 (0.5)	2.6 (0.5)
	$B1_{T=1}$	2.8 (0.3)	4.3 (1.2)	4.3 (1.2)	2.6 (0.6)	3.3 (0.3)	3.3 (0.3)	2.5 (0.2)	3.4 (0.3)	3.4 (0.3)
	$C_{T=0}$	4.7 (0.4)	6.5 (0.6)	4.6 (0.6)	4.2 (0.3)	6.0 (0.7)	4.2 (0.7)	3.9 (0.5)	5.2 (0.7)	4.0 (0.7)
	$C_{T=1}$	8.4 (0.6)	8.3 (1.9)	4.1 (1.6)	7.5 (0.6)	8.3 (1.4)	4.8 (2.2)	7.8 (1.1)	6.2 (1.0)	3.7 (1.5)

Table 8 : Activation Gliding Forces, Pistons A, B2 and C

Silicone/internal surface of container		4 µg/cm²			4 µg/cm²			4 µg/cm²		
Silicone/piston		5 µg/cm²			15µ/cm²g			50µg/cm²		
		Force (N)								
Force (N)		B	S	F	B	S	F	B	S	F
Piston	$A_{T=3}$	13.9 (2.4)	11.1 (4.5)	3.1 (1.0)	17.2 (2.0)	13.5 (4.3)	3.5 (1.7)	14.2 (2.9)	6.7 (2.0)	2.8 (1.0)
	$B2_{T=3}$	2.5 (0.2)	3.5 (0.4)	2.6 (0.3)	2.5 (0.3)	3.4 (0.2)	2.2 (0.3)	2.4 (0.2)	3.3 (0.3)	2.4 (0.3)
	$C_{T=3}$	9.6 (1.4)	9.2.(5.5)	4.1 (1.5)	10.0 (1.4)	8.7 (5.0)	3.7 (1.5)	8.4 (2.1)	6.3 (2.7)	3.3 (0.8)

As Table 7 shows, with the 3 µm thick Parylene C coating according to the invention (piston B1), the friction forces B, S and F are always below 4 N, regardless of the amount of silicone coating added onto the piston 3. The results obtained with a comparative Parylene C coating of a lesser thickness of 0.5 µm (piston C) are markedly inferior. After one month of ageing, the friction forces B and S increase, in the case of comparative pistons A and C, whereas they remain practically unchanged and below 4 N in the case of inventive piston B1.

As shown by Table 8, after three months of ageing, the friction forces B and S continue to increase in the case of comparative pistons A and

C, whereas with piston B2, these same friction forces B and S remain unchanged and below 4 N thanks to the coating 8 according to the invention.

Example 5

The protocol of Example 2 was repeated using a different glass syringe body or glass container 2, which was not coated with an internal film of silicone oil. By contrast, a silicone oil was coated on the pistons prior to assembly or fitting.

The results according to Table 9 were then obtained.

Table 9

Silicone/internal surface of container		0 µg/cm²			0 µg/cm²		
Silicone/piston		15 µg/cm²			50 µg/cm²		
Force (N)		B	S	F	B	S	F
Piston	$A_{T=1}$	29.3 (2.7)	32.0 (6.3)	56.6 (8.1)	31.8 (2.6)	13.0 (1.9)	20.3 (4.5)
	$B1_{T=1}$	9.5 (1.1)	8.8 (1.1)	15.2 (1.5)	9.0 (1.0)	7.0 (1.0)	9.0 (1.0)
	$B2_{T=1}$	7.2 (1.9)	7.7 (0.9)	13.3 (1.0)	8.5 (1.5)	7.4 (1.6)	10.2 (2.4)

As can be seen from Table 9, it seems difficult to make the pistons A move inside the syringe body 2. However, thanks to the 3 µm thick Parylene C coating 8 according to the invention, in the case of pistons B1 and B2, all the friction forces B, S and F are reduced in comparison to piston A, by about a factor of 4 in the case of an additional silicone coating of 15 µg/cm^2 of silicone, and about a factor of 2 in the case of an additional silicone coating of 50 µg/cm^2 of silicone.

Example 6

The protocol of Example 2 was repeated for both of the following scenarios:

Scenario 1: a silicone lubricant was deposited and baked onto the internal surface of the syringe body 2, at a rate of 40 µg for a surface area of 10 cm^2, but no silicone was used or sprayed on the pistons 3.

Scenario 2: a silicone lubricant was sprayed onto the internal surface of the syringe body 2 at a rate of 500 µg for a surface area of 10 cm^2, but no silicone was used or sprayed on the pistons 3. The results obtained are collated in Table 10 below.

Table 10

		Scenario 1			Scenario 2		
Silicone/internal surface of syringe		4 µg/cm²	4 µg/cm²	4 µg/cm²	50 µg/cm²	50 µg/cm²	50 µg/cm²
Silicone/piston		0 µg/cm²	0 µg/cm²	0 µg/cm²	0 µg/cm²	0 µg/cm²	0 µg/cm²
Force (N)		B	S	F	B	S	F
Piston	$A_{T=0}$	6.6 (0.3)	6.9 (1.4)	4.0 (1.4)	5.5 (0.5)	1.2 (0.3)	4.0 (2.0)
	$A_{T=1}$	15.7 (2.9)	5.3 (2.6)	6.1 (4.2)	8.6 (1.1)	1.6 (0.7)	5.6 (4.1)
	$B1_{T=0}$	2.1 (0.1)	2.5 (0.3)	2.6 (0.3)	1.9 (0.2)	1.3 (0.3)	2.1 (0.7)
	$B1_{T=1}$	3.0 (0.4)	3.4 (0.5)	2.8 (0.6)	2.2 (0.2)	1.4 (0.3)	2.4 (0.6)
	$C_{T=0}$	3.9 (0.6)	6.6 (2.5)	3.9 (2.5)	4.2 (0.6)	1.0 (0.4)	4.7 (2.9)
	$C_{T=1}$	14.4 (2.2)	4.8 (2.1)	3.6 (1.1)	5.4 (1.2)	1.3 (0.5)	4.3 (2.8)
	$A_{T=3}$	17.2 (6.1)	4.3 (2.4)	2.9 (1.2)	10.0 (1.0)	1.5 (0.3)	4.0 (3.0)
	$B2_{T=3}$	2.8 (0.4)	3.4 (0.3)	2.7 (0.4)	2.3 (0.2)	1.5 (0.2)	2.2 (0.4)
	$A_{T=6}$	20.5 (4.0)	6.1 (3.0)	3.0 (1.0)	15.1 (1.4)	2.5 (1.5)	3.0 (2.0)
	$B2_{T=6}$	2.3 (0.5)	2.7 (0.5)	2.6 (0.6)	2.0 (0.5)	1.6 (0.3)	3.2 (1.0)

With pistons A and C, the friction forces B, S and F were relatively high, something which does not appear to be acceptable for a medical device. Conversely, with pistons B1 and B2 which are provided with the coating 8 of the invention, the friction forces B and F were entirely compatible with the way in which a medical device 1 is used.

After one month of ageing, the friction forces B, S and F for pistons A and C had increased appreciably, especially the friction forces B. Conversely, the friction forces B, S and F of pistons B1 had increased very little.

After three months and six months of ageing, all the friction forces B, S and F measured for piston B2 had remained lower than 3.5 N. Using the invention, it therefore appears to be possible, thanks to the coating 8 according to the invention, to eliminate the use of silicone oil on rubber pistons 3 in medical devices 1.

Additional tests similar to the tests described above demonstrated that the optimum mean thickness for the coating 8 for the medical device 1 of the invention ranges between 3 and 10 μm and preferably from 3 to 5 μm.

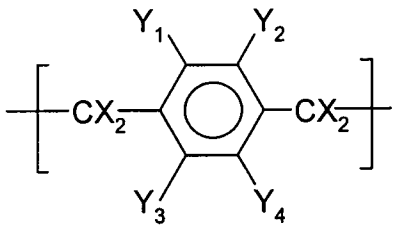
In the examples described, containers 2 are syringe barrels made of glass. Of course, the invention is not limited to glass containers and also includes containers made of plastic, polymer and any other suitable materials.

In an alternative form of embodiment that has not been illustrated, the coating according to the invention is on the container rather than on the piston. In this configuration, the piston may be provided with a coating of silicone to face the coating at the contact region.

In an embodiment of the invention, an adhesion-promoting layer that encourages the coating to bond with the container 2 may be provided.

CLAIMS

1. Medical device (1) comprising at least a first part (2; 3) and a second part (3; 2), said first and second parts (2, 3) being able to move one relative to the other and determining between them at least one contact region (10), at least one of said first and second parts (2, 3) being provided with at least one coating (8) designed to at least encourage the relative sliding of said first and second parts (2, 3) one relative to the other and tightness in said contact region (10), and said coating (8) consisting of at least one polymer material comprising polymer chains having the following repeat unit:



in which X represents a halogen, for example F, or a hydrogen,
and in which Y_1, Y_2, Y_3, Y_4 each independently represent a halogen, for example Cl, or a hydrogen,
characterized in that the mean thickness of said coating (8) ranges from 3 to 10 μm.

2. Medical device (1) according to Claim 1, characterized in that the mean thickness of said coating (8) ranges from 3 to 5 μm.

3. Medical device (1) according to Claim 1, characterized in that said first part (2) is a container intended to accommodate a medical product (6) and in that said second part (3) is a piston movable in said container (2) in order to vary the volume of said medical product (6) contained in said container.

4. Medical device (1) according to Claim 1, characterized in that said second part consists of a viscoelastic material designed to encourage tightness at said contact region.

5. Medical device (1) according to Claim 1, characterized in that said first part (2) is a container intended to accommodate a medical product (6), said medical device (1) comprising a piston movable in said container (2), said second part (3) being an intermediate part (9) located between said container and said piston.

6. Medical device (1) according to Claim 3, characterized in that said coating (8) is provided at least on said container (2).

7. Medical device (1) according to Claim 3, characterized in that said coating (8) is provided at least on said piston (3).

8. Medical device (1) according to Claim 5, characterized in that said coating (8) is provided at least on said intermediate part (9).

9. Medical device (1) according to Claim 1, characterized in that said coating (8) is continuous and elastic.

10. Medical device (1) according to Claim 1, characterized in that said polymer material is chosen from the group consisting of poly(p-xylylene), poly(p-meta-chloroxylylene), poly(p-ortho-chloro/meta-chloroxylylene) and poly(p-difluoroxylylene).

11. Medical device (1) according to Claim 1, characterized in that said contact region (10) further includes a lubricant other than said coating (8).

12. Medical device (1) according to Claims 6 or 7 or 8 and 11, characterized in that said coating (8) provided on said container (2) or on said piston (3) or on said intermediate part (9) is at least partially covered with said lubricant.

13. Medical device (1) according to Claims 6 or 7 or 8 and 11, characterized in that said piston (3) or said container (2) or said intermediate part (9), not provided with said coating (8), is at least partially covered with said lubricant.

14. Medical device (1) according to Claim 11, characterized in that said lubricant contains silicone.

15. Medical device (1) according to Claim 1, characterized in that said coating (8) is designed to have at least one outer surface relative to which at least one of said first and second parts (2, 3) is intended to move, said outer surface having a mean roughness Ra of less than 2.5 µm, preferably less than 2 µm and, for example, of the order of 1.0 µm.

16. Medical device (1) according to Claim 1, characterized in that said polymer material consists of poly(p-meta-chloroxylene).

17. Medical device (1) according to Claim 1, characterized in that it includes an injection device.

18. Medical device (1) according to one of claims 1-17, characterized in that said first part (2) is made out of glass.

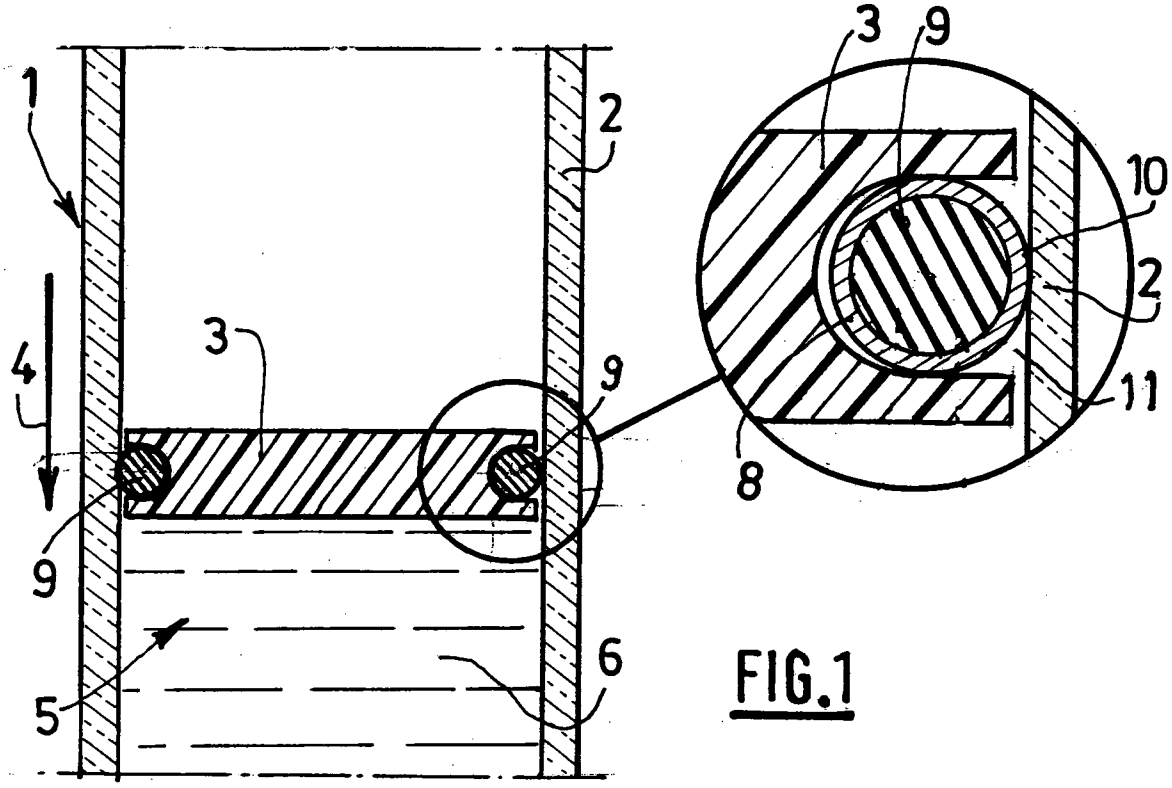


FIG.1

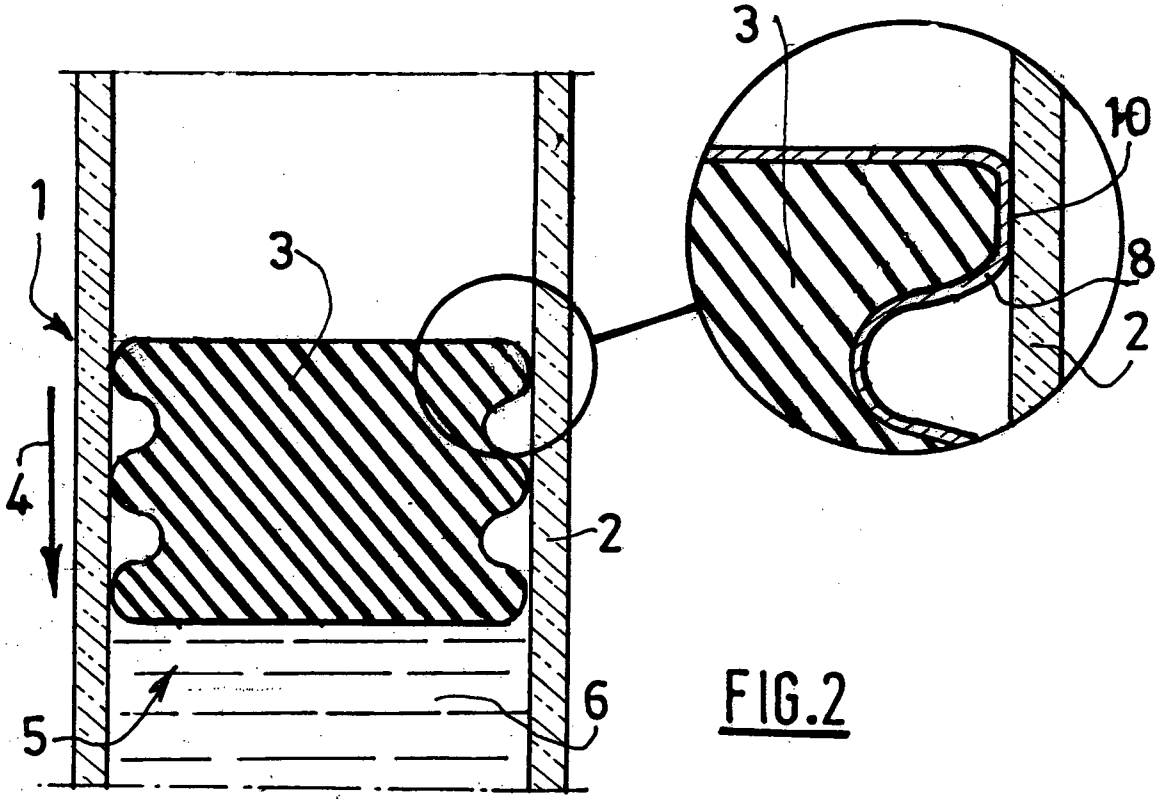


FIG.2

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2007/003339

A. CLASSIFICATION OF SUBJECT MATTER
INV. A61L31/10 A61M5/315

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)
A61L A61M

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	JP 2005 160888 A (TERUMO CORP) 23 June 2005 (2005-06-23) abstract; figure 1	1-18
X	US 5 064 083 A (ALEXANDER BARBARA [US] ET AL) 12 November 1991 (1991-11-12) column 4, line 60 - line 64; figure 3	1
Y	US 2 735 735 A (MARTIN S. ABEL) 21 February 1956 (1956-02-21) column 3, line 69 - column 4, line 4; figure 3	1-18
Y	WO 01/27210 A (INNOVATA BIOMED LTD [GB]; BRAITHWAITE PHILIP [GB]) 19 April 2001 (2001-04-19) abstract; claims 28,29	1-18

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
15 May 2008	27/05/2008

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2007/003339

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A	US 2 842 127 A (JAMES EVERETT SAMUEL) 8 July 1958 (1958-07-08) figures	1-18
A	US 4 973 504 A (ROMBERG VAL G [US] ET AL) 27 November 1990 (1990-11-27) column 1, line 5 - line 11; figures	1
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