United States Patent

[11] **3,552,441**

[72] Inventor **Hartmut Luhleich**
Geesthacht, Germany (Chlodiwigplatz 15
516 Duren Germany)

[21] Appl. No. **670,600**
[22] Filed **Sept. 26, 1967**
[45] Patented **Jan. 5, 1971**

[54] **PIERCABLE CLOSURE DIAPHRAGM FOR A CHAMBER**
18 Claims, 25 Drawing Figs.

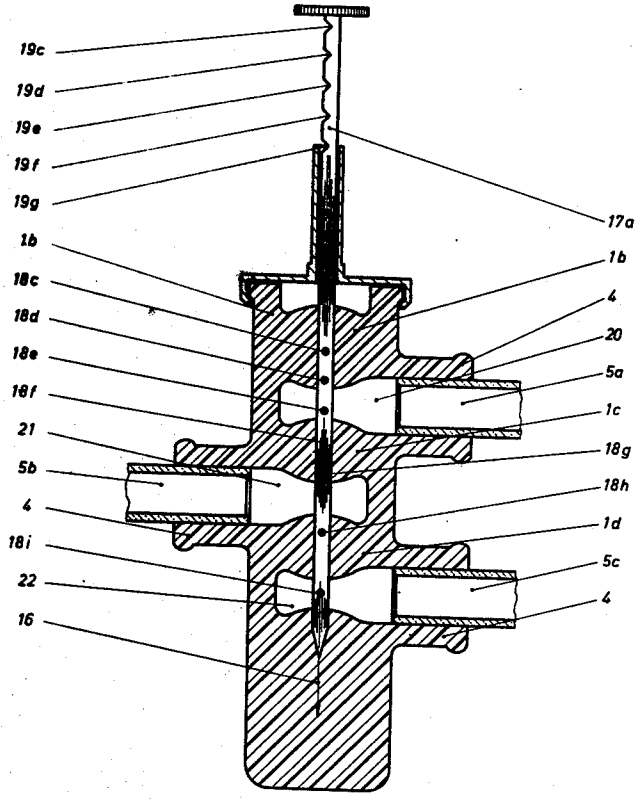
[52] **U.S. Cl.** **137/625.48,** 137/318, 128/272
[51] **Int. Cl.** **F16k 11/07**
[50] **Field of Search** ..... 128/218.1, 218.M, 272; 251/Needle Valve; 137/525.1, 318, 625.68, 625.4, 625.48; 141/329

[56] **References Cited**
UNITED STATES PATENTS

1,509,916	9/1924	Waite	128/272X
2,020,828	12/1935	Goldberg	128/218.1
2,193,489	3/1940	Nevin	128/218.1
2,653,607	9/1953	Deans	128/218.M
2,670,871	3/1954	Spiess et al.	128/272X
2,837,093	6/1958	Tash	128/218.1
2,854,998	10/1958	MacGlashan et al.	251/284X
3,465,774	9/1969	Kautz et al.	137/81.5

Primary Examiner—M. Cary Nelson
Assistant Examiner—Michael O. Sturm
Attorney—Waters, Roditi, Schwartz & Nissen

ABSTRACT: A diaphragm having opposite surfaces of convex shape, in the manner of a lens, and constituted of elastic material to enable its being pierced by the sharp end of a hollow element and form a seal with the hollow element during penetration and withdrawal thereof, whereby the diaphragm can constitute a closure for an open vessel and prevent contamination of its interior.



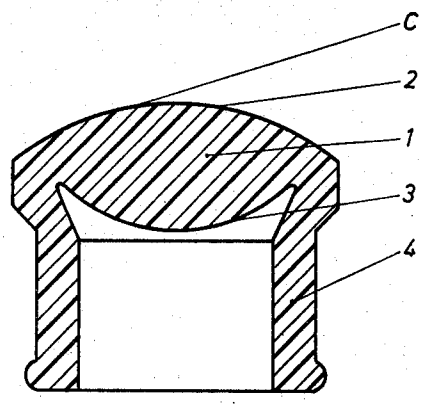
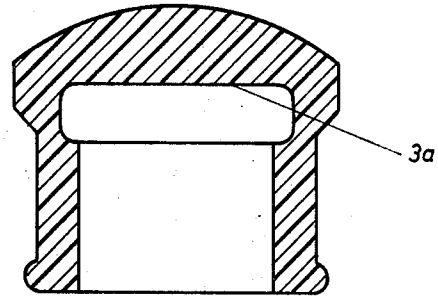
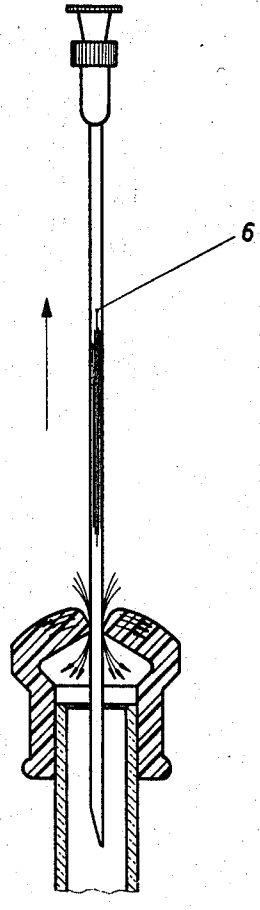


Fig. 1



PRIOR ART

Fig. 2



PRIOR ART

Fig. 3

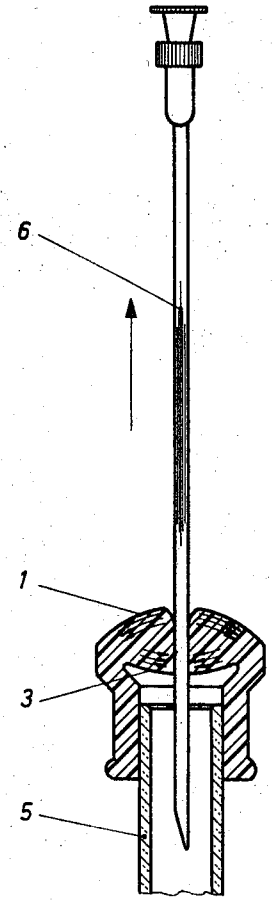


Fig. 4

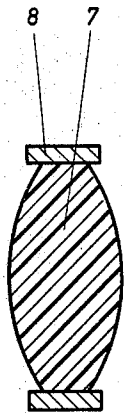


Fig. 5

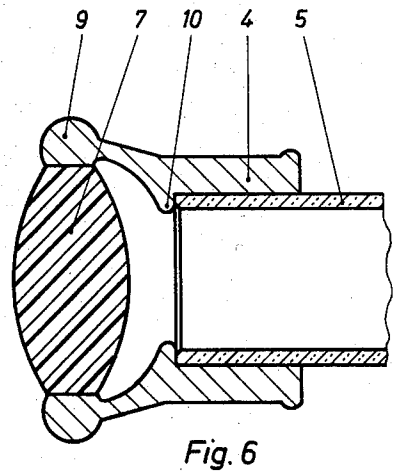


Fig. 6

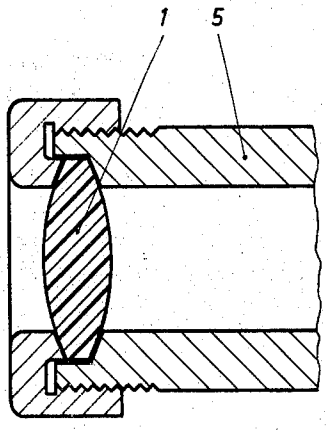


Fig. 7

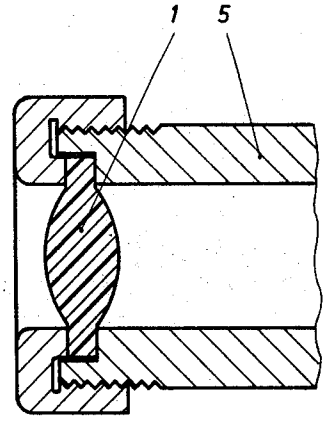


Fig. 8

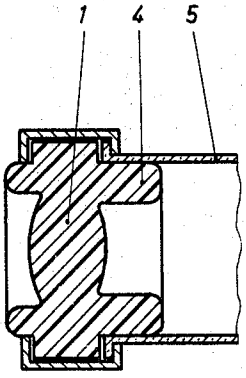


Fig. 9

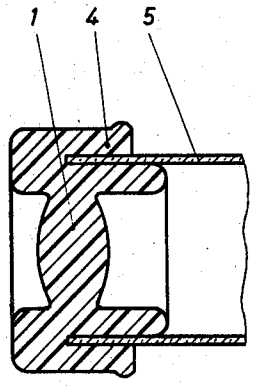


Fig. 10

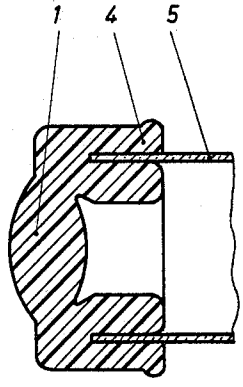


Fig. 11

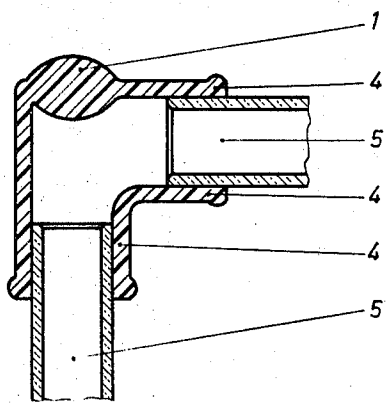


Fig. 12

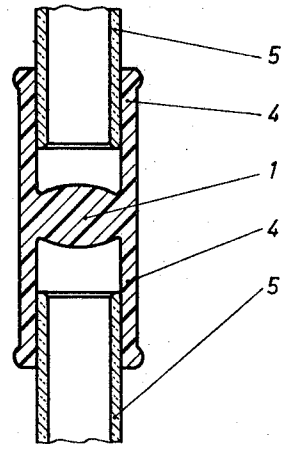


Fig. 13

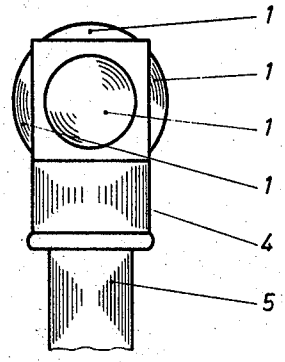


Fig. 14

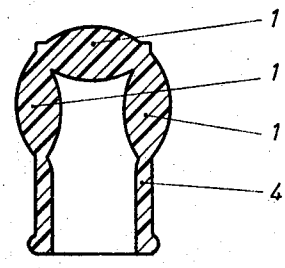


Fig. 15

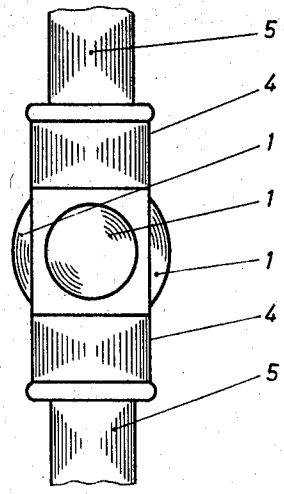


Fig. 16

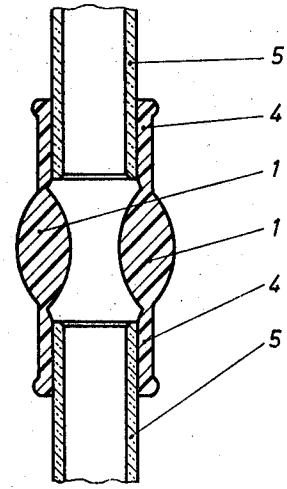


Fig. 17

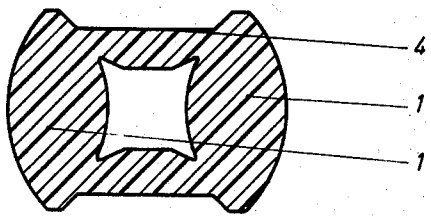


Fig. 18

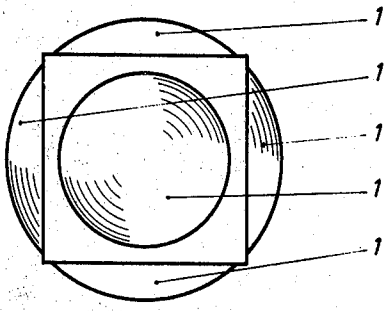


Fig. 19

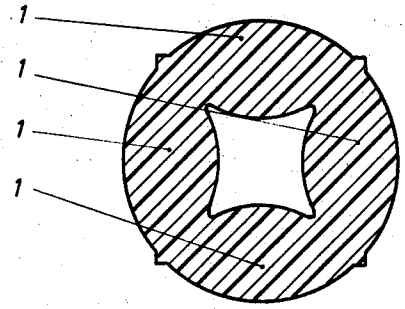


Fig. 20

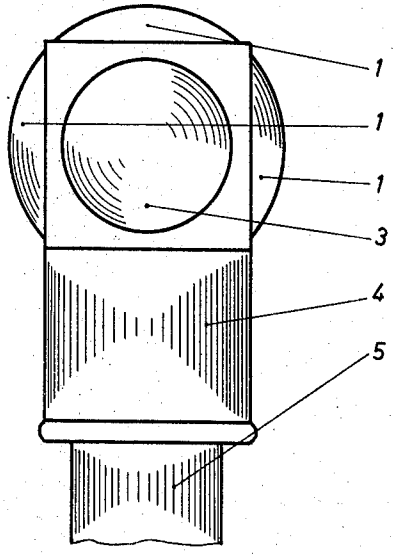


Fig. 21

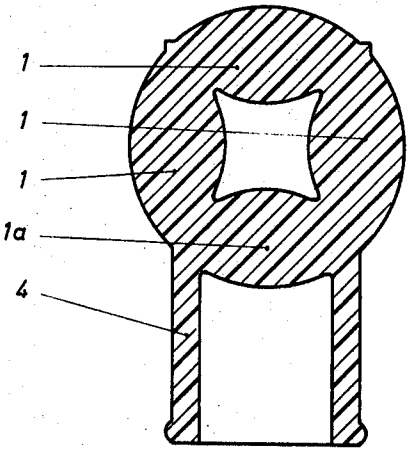


Fig. 22

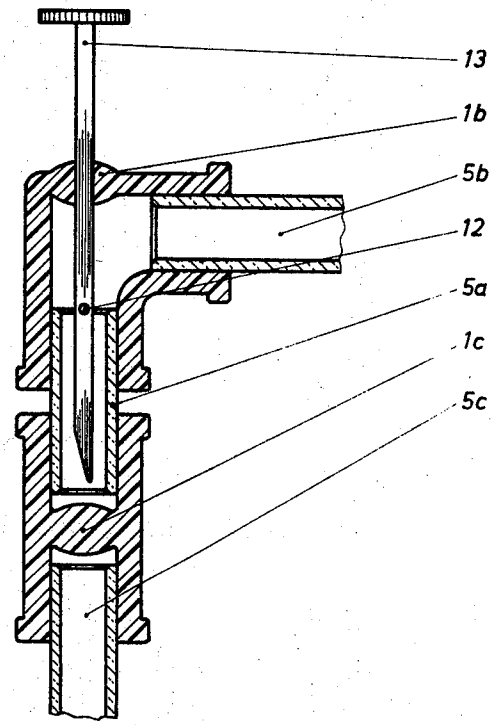


Fig. 23

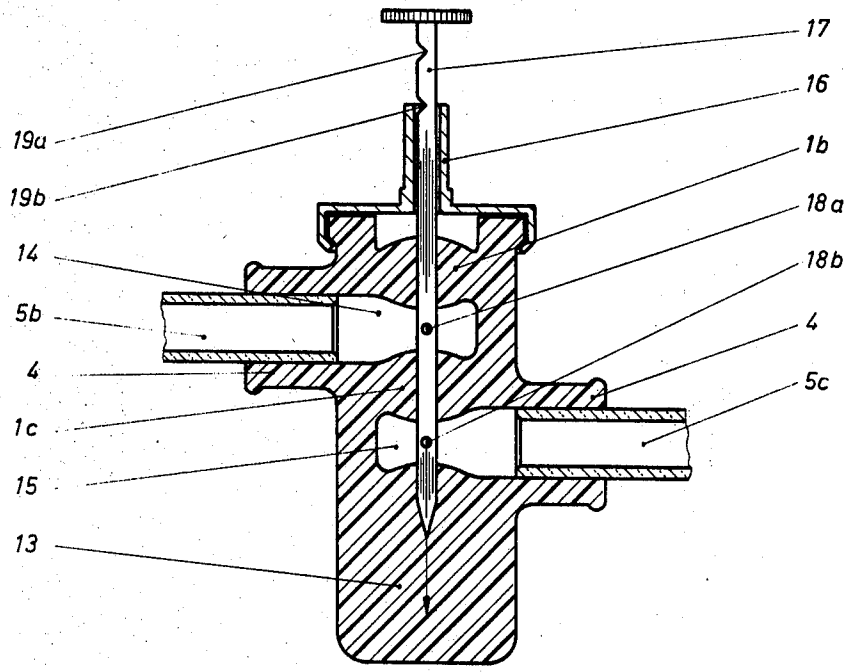


Fig. 24

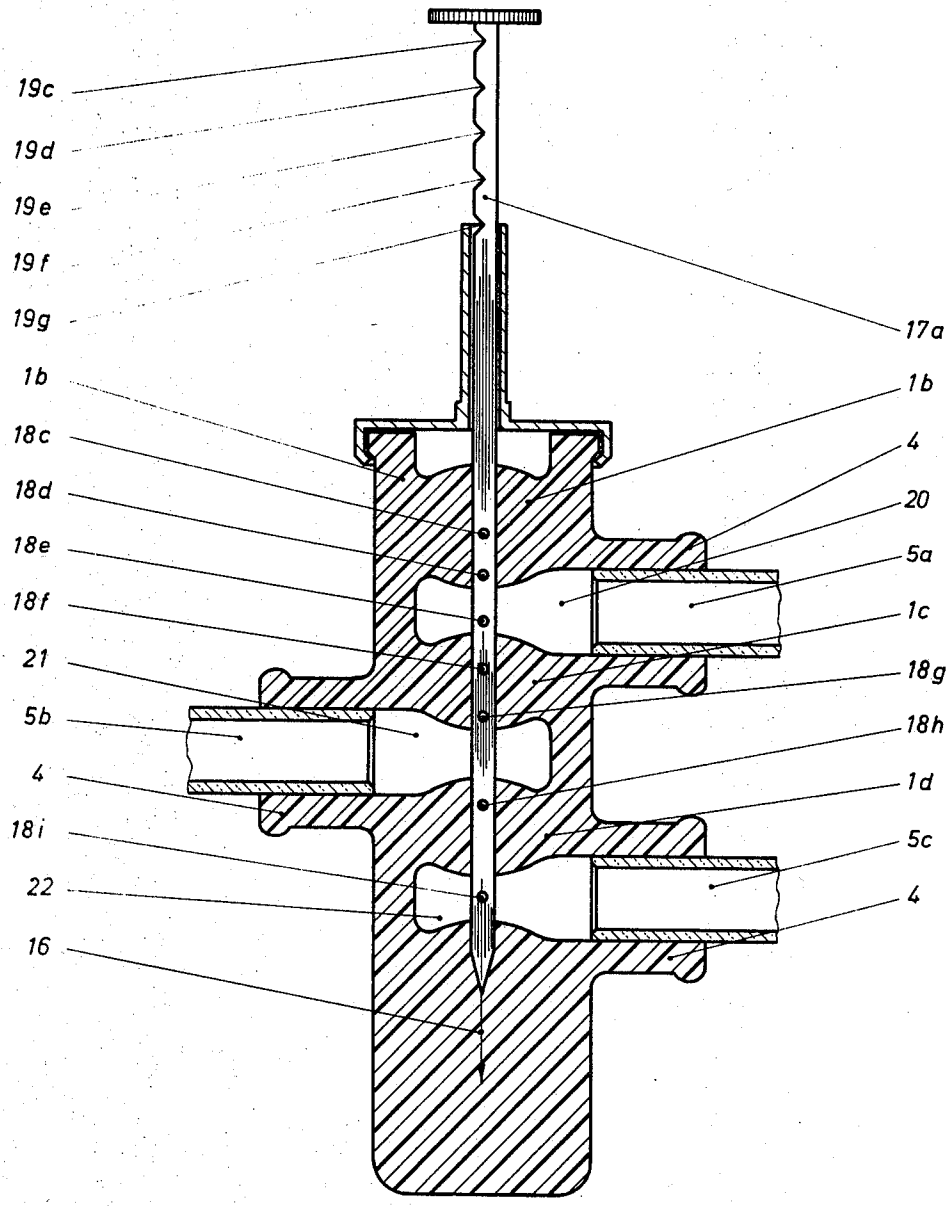

19c
19d
19e
19f
19g
1b
18c
18d
18e
18f
21
5b
4
18i
22
16
17a
1b
4
20
5a
1c
18g
18h
1d
5c
4

Fig. 25

PIERCABLE CLOSURE DIAPHRAGM FOR A CHAMBER

BRIEF SUMMARY OF THE INVENTION

The invention relates to pierceable diaphragms for closures of vessels such as ampuls flasks, cuvettes, or other chemical or physical apparatus, as well as to parts of chemical or physical apparatus provided with pierceable diaphragms such as prefabricated valves provided with such diaphragms. Pierceable caps, preferably of rubber, with a plane or convex diaphragm for closing ampuls, flasks or nipples of chemical apparatus for operations in closed systems are known (see Journal Kerntechnik, Isotopentechnik, and Chemie, Vol. 7, 1965, No. 7, p.p. 326—329). They are applied with a sleeve over the neck of the vessel opening and permit filling or withdrawing substances by means of hypodermic needles. Such diaphragms provide a reliable seal during the piercing of the needle. By carrying along material of the diaphragm due to friction therewith, the curvature of the diaphragm is flattened and the diaphragm surface thus reduced, so that the material of the diaphragm close to the surface is compressed around the needle. When the needle is extracted, this effect is reversed, however, since the diaphragm is curved to the outside beyond its rest position, due to the friction on the needle, so that the edges of the piercing point gape, resulting in leaks. The latter are particularly evident when the vessel is under pressure. Such diaphragms thus do not provide a reliable seal.

Pierceable diaphragms of elastic material have also been suggested (German utility model No. 1,928,384) which are curved in the case of substantially parallel diaphragm surfaces toward the vessel interior. In this arrangement a leak by gaping of the edges is avoided when the needle is extracted, but the leakage effect is present during the piercing of the diaphragm by the needle.

In known diaphragms which are already pierced, leaks appear due to pressure differences between the chambers separated by them, since the piercing openings are stretched apart due to deformation of the diaphragm. In curved diaphragms this is avoided when the pressure acts on the convex side, but the leaks are more aggravated if the direction of the pressure is reversed, where the piercing holes act like valves.

An object of the invention is to provide diaphragms which avoid the known disadvantages and which seal reliably even after several punctures both during the piercing and the extraction of the needle and in the case of pressure differences between the separated chambers.

This objective is achieved according to the invention by the provision of a convex curvature on the opposite surfaces of the diaphragm, in the manner of a lens. The diaphragm according to the invention consists of a sufficiently elastic material which restores deformation immediately. Due to its convex form on both sides, the piercing point is sealed automatically both during the piercing and during the extraction of the needle, due to the material of the diaphragm carried along by friction, the movement of the needle causing flattening and inward deformation of the convexity of the diaphragm opposing the direction of motion of the needle whereby a reliable seal is achieved by the resulting compression of the surface material around the needle.

In the diaphragm according to the invention no leaks are formed in the case of pressure differences between the adjoining chambers, since the outside curvature of the diaphragm positively prevents gaping of the piercing holes. The pressure acts in the diaphragm according to the invention on a curvature opposing the direction of the pressure, so that the piercing openings are compressed and reliably sealed.

It is of advantage to arrange the diaphragm according to the invention so that it is under inward radial pressure at its periphery in order to obtain an initial stress which improves the elastic restoration of the piercing holes. Diffusion of gases through the diaphragm is thus prevented even after several punctures and even if there is a major pressure difference between the chambers separated by the diaphragm. This pressure effect on the diaphragm can be achieved by mounting the diaphragm in a ring which is constituted of a firm or elastic material and whose inside diameter is less than the outside diameter of the diaphragm body. The diaphragm body is therefore inserted under pressure into the ring. The joint between the ring and the diaphragm body can be formed by cementing, welding or a toothing, as for example, by corrugations.

In this arrangement it is possible to use softer rubber as a material for the diaphragm, which by themselves would cause leaks in their connection with the wall of the chambers to be closed by the diaphragm, but which are better suitable to close the piercing damages both during the stress by piercing and in the long run.

This arrangement is also particularly suitable for sealing ducts of needles or wires extending through the diaphragm and which are to remain therein for a longer period of time.

The diaphragm according to the invention is secured in known manner by elastic sleeves or collars rigidly connected with the diaphragm and pulled over the open neck of the vessels or by screwing or pressing.

The diaphragms according to the invention can also be attached on or in pipe lines, for example, for chemical apparatus or on or in sleeves connecting tubes inserted therein.

The compressed diaphragms in the above-mentioned ring can be secured together with the ring in the same manner as described above. However, the diaphragm bodies can also be inserted under stress in the collars, sleeves or tubes themselves, which then assume the function of the above-described ring.

In order to avoid leaks after several punctures, several diaphragms according to the invention can be arranged on the same opening side by side. The diaphragm can be formed in walls defining a polyhedron, such as a cube, and a connecting collar extends from the polyhedron.

The diaphragms according to the invention can also be used in the known protective tubes (see Dr. H. Luhleich: "On Isomers in the Silane-series on the Basis of Preparative and Physical-Chemical Investigations," Cologne, Diss, 1958, p. 55, journal: Zeitschrift f. inoganische and allegemeine Chemie, Vol. 303, 1960, p.p. 294—302 and journal: Zeitschrift f. Naturforschung, Vol. 14*b*, No. 7, 1955, p.p. 466 and 467). The protective tube can be designed preferably as a closed hollow body where opposing walls include diaphragms according to the invention, particularly in the form of a cube, in each of the six faces of which is provided a diaphragm.

In a corresponding manner the diaphragms according to the invention can also be used in the known sealing lock technique (see German utility model No. 1,920,471) which serves to improve the sealing of pierceable cap closures. Instead of two superposed piercing caps, a hollow body can be arranged ahead of the diaphragm directly sealing the opening to be sealed, in the wall of which are provided several diaphragms according to the invention side by side.

In another embodiment of the invention valves can be formed by using the diaphragms according to the invention. In the walls of the valve chambers, namely between the latter and the outside atmosphere, there are arranged diaphragms according to the invention in series in parallel planes in such a way that they can be pierced jointly by a needle. In a wall of the needle penetrating them in this manner, are provided lateral openings by which the chambers of the valves are connected with each other selectively, depending on the depth of puncture of the needle. Such valve arrangements can be composed of a one piece body with integral diaphragms according to the invention, or they can consist of a uniform body provided with pipe connection sleeves.

Finally, chemical or physical apparatus can be assembled easily and without difficulty so that they can be exchanged and used again from single elements carrying diaphragms according to the invention together with pipe connection, angle and reducing sleeves with standardized connection cross sections

of rubber or other elastic material together with other standardized elements, pipe lines and devices according to the assembly system.

If diaphragms according to the invention are exposed to corrosive substances or to higher temperatures, surface coatings, for example of inert metals or any other laminated structure may be of advantage.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a sectional view of a pierceable cap with a diaphragm according to the invention;

FIG. 2 is a sectional view of a known cap with curved diaphragm;

FIG. 3 is a sectional view of the known cap according to FIG. 2 during the extraction of a needle;

FIG. 4 is a sectional view of the pierceable cap with the diaphragm according to the invention during the extraction of the needle;

FIG. 5 is a sectional view of another embodiment of the diaphragm according to the invention;

FIG. 6 is a sectional view of a pierceable cap with the diaphragm according to the invention inserted therein;

FIGS. 7 and 8 are sectional views of diaphragms secured on the open neck of a vessel;

FIG. 9 is a sectional view of a diaphragm according to the invention which is secured under pressure with a flanged sheet metal ring on the open neck of a vessel;

FIGS. 10 and 11 are sectional views of diaphragms according to the invention, which are connected with integral fastening collars mounted on the open neck of a vessel;

FIG. 12 is a sectional view of a diaphragm according to the invention arranged in the wall of an elbow pipe connection member;

FIG. 13 is a sectional view of a pipe connection member with a diaphragm according to the invention arranged transversely therein;

FIG. 14 is a side view of a pierceable cap with five walls with diaphragms according to the invention;

FIG. 15 is a sectional view of the pierceable cap of FIG. 14;

FIG. 16 is a side view of a pipe connection member with four walls having diaphragms according to the invention arranged therein;

FIG. 17 is a sectional view of the pipe connection member of FIG. 16;

FIG. 18 is a sectional view of a protective tube with two diaphragms according to the invention in the form of a closed hollow body;

FIG. 19 is a side view of a protective tube in the form of a cubic closed hollow body with six diaphragms according to the invention;

FIG. 20 is a sectional view of the protective tube of FIG. 19;

FIG. 21 is a side view of a sealing lock with five outside diaphragms according to the invention and one inside diaphragm according to the invention;

FIG. 22 is a sectional view of the sealing lock of FIG. 21;

FIG. 23 is a sectional view through a two-way valve composed of a connection member according to FIG. 12 and one according to FIG. 13;

FIG. 24 is a sectional view of a two-way valve according to the invention; and

FIG. 25 is a sectional view of a three-way valve according to the invention.

DETAILED DESCRIPTION OF THE INVENTION

In FIG. 1 there is shown a cap C with a lens-shaped diaphragm 1 provided with a convex outer surface 2 and with a convex inner surface 3. A connection sleeve 4 extends from the diaphragm and can be attached to a vessel or tube 5 with a contact pressure sufficient for sealing the vessel or tube 5 (FIG. 4). The diaphragm 1 and sleeve 4 are integral and composed of elastic material, as for example, rubber.

FIG. 2 shows for comparison purposes a known pierceable cap with a diaphragm having a planar inner surface 3*a*. The caps are adapted for being placed on vessels such as flasks, ampuls, tubes etc. for closing the open mouths thereof. In order to remove or introduce a substance in the vessel, a hollow tubular element such as a hypodermic needle is inserted into the vessel through the diaphragm. Such hollow tubular elements will hereafter be referred to simply as a tubule.

FIG. 3 shows the extraction of a tubule 6 from the known cap with the diaphragm with the plane surface 3*a*. Since the material of the diaphragm is pulled upwardly with the tubule due to the frictional engagement with the tubule, the region where the tubule pierces the cap is caused to widen externally thereby exposing an opening for the penetration into the vessel of impurities from the outside atmosphere.

FIG. 4 shows how the material at the surface 3 of the diaphragm according to the invention is compressed around the tubule by the removal of the tubule so that the danger of leakage is positively prevented. During the piercing of the cap with the tubule, the process is correspondingly reversed. Thus, the diaphragm according to the invention is capable of sealing the vessel both during insertion and removal of the tubule from the diaphragm.

In FIG. 5 the diaphragm body 7 is inserted into a tension ring 8 whose inside diameter is smaller than the outside diameter of the normal diaphragm body 7. Consequently, the diaphragm is compressed by its insertion into the tension ring 8. Thereby, the diaphragm is subjected to an initial radial stress which tends to close any openings formed in the diaphragms thus maintaining the interior of the vessel isolated from the outside atmosphere.

The ring 8 can consist of rigid or elastic material. In the latter case, the inside diameter of the ring must be somewhat smaller to compensate for its expansion. The diaphragm body 7 consists of a relatively soft rubber, which closes the piercing openings automatically since it is under initial stress by the pressure of the ring.

In the cap represented in FIG. 6, an annular bulge 9 at the upper edge of the collar 4 performs the function of the tension ring 8. Its inside diameter, before insertion of the diaphragm body 7, is likewise smaller than the outside diameter of the diaphragm body. When the diaphragm body 7 is inserted into the ring 9, the upper part of the collar 4 is widened. In order to avoid loosening of its seat on the neck 5 of the vessel, an annular internal projection 10 constituting a stop is provided on the inside of the collar 4 which limits penetration of the neck 5 and at the same time protects the penetrating point of the tubule against damage by contact with the edge of the vessel neck 5. The material of the diaphragm body 7 is softer than the rubber of which the collar 4 is made.

FIGS. 7—11 show different known means of fastening the diaphragm according to the invention on the neck of the vessel 5. In FIGS. 7 and 8 the diaphragm is secured by screwing, in FIG. 9 by flanging and in FIGS. 10 and 11 by insertion of the neck in an annular groove provided in the collar 4.

FIGS. 12 and 13 show diaphragms 1 formed in vessel connection elements having collars 4 for receiving the ends of the vessels 5 to be connected. In FIG. 12 the diaphragm 1 is formed in a wall of the connection element whereas in FIG. 13 the diaphragm 1 extends transversely between sleeves 4 which engage the vessels 5.

FIGS. 14 and 15 show a cap of rubber or corresponding elastic material, wherein diaphragms 1 are provided on five sides in cubic arrangement. It thus becomes possible to puncture the cap in the walls of all five sides, thereby enhancing considerably the use and longevity of the cap.

FIGS. 16 and 17 show a vessel connection element with diaphragms 1 according to the invention in corresponding multiple arrangement, and wherein collars 4 are mounted on tube ends 5. Here again, all of the walls with the diaphragms may be pierced thereby prolonging the use of the connection element.

In the embodiment represented in FIG. 18, two opposing diaphragms according to the invention are combined to form a uniform hollow body. In the embodiment according to FIGS. 19 and 20, this hollow body is in the shape of a cube in each wall of which, is provided a diaphragm 1. FIGS. 21 and 22, show a sealing cap which corresponds in its arrangement to the protective tube according to FIG. 19, there being provided a depending collar 4 rigidly connected with the cube for attachment on a vessel 5.

In FIG. 23 the embodiments according to FIGS. 12 and 13 are connected by a tubular piece 5*a* to form in combination with a tubule 11 a valve arrangement, as will be described hereafter. By inserting the tubule 11 through the diaphragms 1*b* and 1*c* a passageway is formed between the ends of vessels 5*b* and 5*c* via a lateral opening 12 in the tubule wall. This passageway is closed, when the point of the tubule is retracted in or withdrawn from diaphragm 1*c*.

In FIG. 24 there is shown a two-way valve in open position. The valve is constituted by a body 13 made entirely of elastic material and a tubule 17. The body 13 which preferably has a round cross section, is provided with two radial connection collars 4 for the ends of vessels 5*c* and 5*b*. Tube 5*b* opens into a cavity 14 in the body 13 and tube 5*c* opens into cavity 15 in the body. Cavity 14 is bounded on opposite sides by the two diaphragms 1*b* and 1*c*. Cavity 15 is bounded on one side by diaphragm 1*c* and on the opposite side by body 13. On the upper surface of the body 13 is a guide 16 for the tubule 17. The latter penetrates axially through the diaphragms 1*b* and 1*c* and its pointed end pierces the body below the cavity 15. The tubule has two lateral openings 18*a* and 18*b* as well as setting marks 19*a* and 19*b* on its shaft. The tubule is solid beyond the openings. The valve is opened by setting the tubule 17 at mark 19*b* by establishing communication between the tubes 5*c* and 5*b* via cavities 14, 15, and lateral openings 18*a* and 18*b* of the tubule 17. When the tubule 17 is set at mark 19*a*, the lateral openings 18*a* and 18*b* are respectively inside the diaphragm 1*c* and body 13 so that communication between the ends of vessels 5*b* and 5*c* is interrupted.

The three-way valve represented in FIG. 25 corresponds in its material and principal arrangement to the valve represented in FIG. 24. In contrast to the latter, however, there are provided three collars for the vessels 5*a*, 5*b*, and 5*c* and corresponding cavities 20, 21 and 22 which are separated from each other by diaphragms 1*c* and 1*d* according to the invention. Lateral openings 18*c*, 18*d*, 18*e*, 18*g*, 18*h*, 18*i*, are provided in the walls of the tubule 17*a* and respective setting marks 19*c*, 19*d*, 19*e*, 19*f*, 19*g*, 19*h*, are formed on the tubule shaft. The tubule is solid beyond the endmost openings 18*c*, 18*i*.

When setting the tubule to mark 19*c*, a passageway is established between tubes 5*b* and 5*c* via the lateral openings 18*d* and 18*g*. When the tubule is set at mark 19*d*, all lateral openings 18*c* to 18*i*, are embedded in the material of the body 13 and the diaphragms 1*b*, 1*c* and 1*d*, so that the valve is closed. When the tubule is set at mark 19*e*, a passageway is established between the tubes 5*a*, 5*b* and 5*c* via lateral openings 18*c*, 18*f* and 18*h*. When the tubule is set at mark 19*f*, a passageway is formed between tubes 5*a* and 5*b* via the lateral openings 18*d* and 18*g*. Finally, when the tubule is set at mark 19*g* a passageway is formed between tubes 5*a* and 5*c* via lateral openings 18*i* and 18 *e*.

The bodies according to the invention, carrying the diaphragms, represented in FIGS. 12 to 25 can be made as a one piece body of elastic material. However, diaphragms 7 of softer elastic material as shown in FIG. 5, can be inserted into these bodies when mounted in a tension ring 8. On the other hand, the mountings or walls of these bodies can assume the function of the tension rings 8, as was described for the annular bulge 9 in FIG. 6.

The embodiments described here can be used with standardized tube cross sections and by means of connection sleeves, angles, reducing or branching pieces together with vessels, ampuls or devices, there can be formed according to the invention an assembly system which can serve as exchangeable and reusable parts for apparatus, such as, for chemical purposes.

The advantages achieved with the invention lie in the reliable sealing, which excludes even traces of impurities, during and after the introduction and withdrawal into or from chambers closed by diaphragms according to the invention, as it is necessary when working in closed systems. Such seals could be obtained heretofore only partially and only by means of complicated measures. Another advantage is the easy exchangeability and the resulting possible combination of the elements equipped with diaphragms according to the invention, but particularly the simplicity of the sealing parts according to the invention, which applies particularly to valves produced with diaphragms according to the invention.

I claim:

1. Apparatus for sealing a chamber, said apparatus comprising a diaphragm constituted of a material which is pierceable by a retractable hollow element for gaining access to the interior of the chamber, said material being elastic and having opposite surfaces, through both of which the hollow element passes, said surfaces in normal, unstressed condition of the diaphragm being of convex shape, and in consequence of the specific material of the diaphragm, urging the diaphragm into sealing contact with the hollow element both during penetration and retraction thereof whereby the interior of the chamber is isolated from the outside atmosphere.

2. Apparatus as claimed in claim 1 comprising a rigid ring, said diaphragm being supported in said ring under pressure, said ring having an inner diameter for receiving the diaphragm which is smaller than the outer diameter of the diaphragm whereby the diaphragm is compressed when mounted in the ring.

3. Apparatus as claimed in claim two wherein said ring includes a depending portion adapted for being mounted on a vessel defining said chamber, and an upstanding portion with a terminal annular bulge defining an opening with said inner diameter for receiving said diaphragm.

4. Apparatus as claimed in claim 3 wherein said ring is constituted of elastic material which is less elastic than that of the diaphragm.

5. Apparatus as claimed in claim 1 comprising a sleeve member integral with said diaphragm and depending therefrom for being mounted on a vessel defining said chamber.

6. Apparatus as claimed in claim 1 comprising a body portion including sleeve members extending from said diaphragm for being mounted on two vessels which are to be coupled together.

7. Apparatus as claimed in claim 6 wherein said diaphragm extends transversely in said body portion, said sleeve members being two in number and extending in opposite directions from said diaphragm.

8. Apparatus as claimed in claim 6 wherein said sleeve members are two in number and extend at right angles to one another to form a connection element, said diaphragm being connected to said sleeve members to form a portion of an outer wall of the connection element.

9. Apparatus as claimed in claim 5 wherein said sleeve member has a groove therein or receiving the end of the vessel.

10. Apparatus as claimed in claim 1 wherein said apparatus comprises a single body which has a plurality of pierceable bounding walls, and includes a diaphragm at each of a number of walls.

11. Apparatus as claimed in claim 10 wherein said walls define a polyhedron, each of the walls being provided with a diaphragm.

12. Apparatus as claimed in claim 11 wherein said polyhedron defines an internal closed chamber.

13. Apparatus as claimed in claim 12 comprising an integral depending sleeve member on the polyhedron for being mounted on a vessel defining said chamber.

14. Apparatus as claimed in claim 1 in combination with said hollow element for constituting a valve for controlling communication between a plurality of vessels, said apparatus including a body with means for engaging a plurality of vessels, said body being provided with cavities in communication with respective vessels, said cavities being arranged in alignment, said body including a plurality of said diaphragms arranged in alignment to bound said cavities, said hollow element having closed ends and extending axially through said diaphragms and having a plurality of axially spaced openings which provide communication between selected cavities depending upon the axial position of the hollow element in the body.

15. The combination as claimed in claim 14 wherein said hollow element includes solid end portions beyond the endmost of said openings.

3 The combination as claimed in claim 15 wherein one of said end portions of said hollow element extends externally of the body, said one end portion including exposed marking means for visually indicating the various positions for said hollow element which provide communication between selected cavities.

17. The combination as claimed in claim 14 wherein said body, said diaphragms, and said means for engaging the vessels, are integral and are formed from a one piece element.

18. Apparatus as claimed in claim 3 comprising an internal annular projection on said ring spaced from said diaphragm for limiting the depth of engagement of the ring on the vessel and for covering the upper edge of the vessel.