(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 4 027 081 A2

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
13.07.2022 Bulletin 2022/28

(21) Application number: **22154522.1**

(22) Date of filing: **15.03.2010**

(51) International Patent Classification (IPC):
F25D 19/00 *(2006.01)* ***F25B 9/14*** *(2006.01)*

(52) Cooperative Patent Classification (CPC):
F25D 19/00; F25B 9/14; F25B 9/145

(84) Designated Contracting States:
AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO SE SI SK SM TR

(30) Priority: **16.03.2009 GB 0904500**

(62) Document number(s) of the earlier application(s) in accordance with Art. 76 EPC:
19187223.3 / 3 620 732
10710389.7 / 2 409 096

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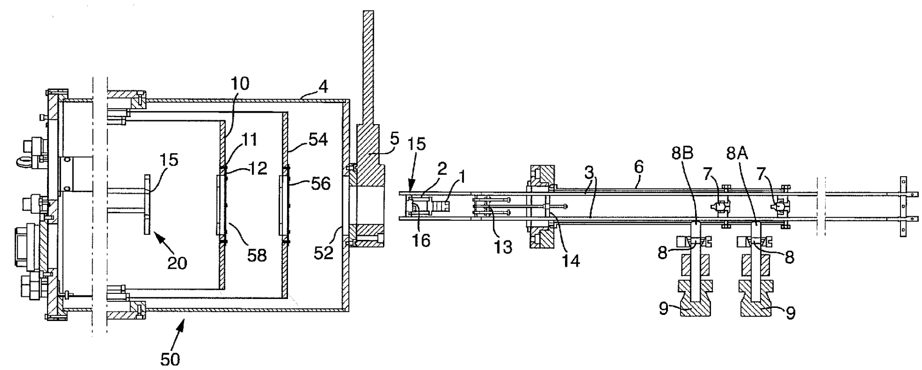
Remarks:
•This application was filed on 01-02-2022 as a divisional application to the application mentioned under INID code 62.
•Claims filed after the date of filing of the application / after the date of receipt of the divisional application (Rule 68(4) EPC).

(54) CRYOGEN FREE COOLING APPARATUS AND METHOD

(57) A cryogen free cooling apparatus comprises at least one heat radiation shield (54) surrounding a working region (20) and located in a vacuum chamber (4). A cryogen free cooling system has a cooling stage coupled to the heat radiation shield (54). Aligned apertures (56,58) are provided in the heat radiation shield and vacuum chamber walls. Sample loading apparatus has a sample holding device (2) attached to one or more elongate probes (3) for inserting the sample holding device through the aligned apertures (56,58) to the working region (20); and a thermal connector enables the sample holding device to be releasably coupled for heat conduction via said connector to a cold body or cold bodies within the vacuum chamber so as to pre-cool a sample on or in the sample holding device.

Fig.1.



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Description

[0001] The invention relates to a cryogen free cooling apparatus and a method for using such an apparatus.

[0002] When operating cryogenic equipment for low temperatures (less than 100 Kelvin) or ultra low temperatures (less than 4 Kelvin), there is often a need to change a sample or other materials at the cold part of the equipment. With conventional equipment using liquid cryogens such as Helium or Nitrogen, this is usually done by warming the equipment up and opening the equipment, or removing a part of the equipment and warming that up. The sample is then changed at room temperature. As this can be a slow process, some conventional cryogenic systems using liquid cryogens are fitted with more rapid sample change mechanisms that allow the majority of the system to remain cold. A key challenge with these systems is that the sample is entered into the equipment at room temperature, typically around 300K and then moved to another position where thermal contact is made with a body at a much lower temperature which in some systems can be lower than 1K. In systems using liquid cryogens the sample and associated mounting and connection equipment is usually pre-cooled either by passing it through cold cryogen gas on its way in to the system or by passing cold cryogen gas or liquid through the sample transfer mechanism, this reduces the thermal shock both on the sample and on the equipment.

[0003] More recently, cryogenic systems that do not require the addition of liquid cryogens or that only require liquid nitrogen during initial cool down have been developed. These are generally known as cryogen free (or "cryofree") systems. These systems use a mechanical cooler such as a GM cooler, Stirling cooler or a pulse tube to provide the cooling power. Because the cooling power of commercially available coolers is somewhat lower than the cooling power available from a reservoir of liquid cryogen, these systems can typically take longer to warm up, change the sample and cool down. There is therefore a considerable need for a method of changing samples in cryogen free systems without the need to warm up the entire system.

[0004] Some examples of known load locks for loading samples into a cryofree cryostat are described in US-A-4446702, US-A-4577465, US-A-5077523, US-A-5727392, US-A-5806319, US-A-5834938, US-A-20070234751 and US-A-20080282710.

[0005] With cryogen free systems there are a number of technical challenges when attempting to load a warm sample in to a cold cryostat. Firstly, the internals of the system are usually contained within a sealed vacuum vessel to reduce heat load. Secondly, within that sealed vacuum vessel, the sample space is usually enclosed by one or more radiation shields to further reduce the heat load. Thirdly, there are no liquid cryogens available to pre-cool the sample as it moves from room temperature to the cold mounting body. Also, electrical contacts need to be remotely made to the sample when it is loaded in the cryostat. This invention seeks to provide solutions to these problems.

[0006] In accordance with a first aspect of the present invention, a cryogen free cooling apparatus comprises at least one heat radiation shield surrounding a working region and located in a vacuum chamber; a cryofree cooling system having a cooling stage coupled to the heat radiation shield; aligned apertures in the heat radiation shield and vacuum chamber wall; sample loading apparatus having a sample holding device attached to one or more elongate probes for inserting the sample holding device through the aligned apertures to the working region; and a thermal connector, whereby the sample holding device can be releasably coupled for heat conduction via said connector to a cold body or cold bodies within the vacuum chamber so as to pre-cool a sample on or in the sample holding device.

[0007] Typically, the sample loading apparatus further includes a vacuum vessel in which the sample holding device and elongate probe are movably mounted, the vacuum vessel being connectable to the aperture of the vacuum chamber wall.

[0008] In accordance with a second aspect of the present invention, a method of loading a sample into the working region of cryogen free cooling apparatus according to the first aspect of the invention comprises:

placing a sample in or on the sample holding device;
securing the vacuum vessel of the sample loading apparatus to the vacuum chamber and aligned with the aperture of the vacuum chamber;
evacuating the vacuum vessel;
opening the aperture of the vacuum chamber and operating the or each elongate probe to insert the sample holding device through the opened aperture so that the sample holding device is thermally coupled to a cold body;
allowing the sample in or on the sample holding device to be cooled as a result of heat conduction to the cold body; and either

a) disconnecting the sample holding device from the cold body; and operating the or each elongate probe to insert the sample holding device into the working region, or
b) strengthening the thermal connection to the cold body to increase heat flow away from the sample and further cool the sample.

[0009] We have devised a new type of apparatus in which the problems set out above are overcome by utilizing a cold body within the vacuum chamber to precool a sample before the sample reaches the working region.

[0010] Although various cold bodies within the cryostat could be used, such as any cold surface coupled to the cooling stage of the cooling system or to an intermediate stage of a sub 4K cooler such as a still of a dilution refrigerator, it is most convenient to utilize the heat radiation shield already present. A further possibility is that the cold body is a body held at the lowest temperature within the cooling apparatus in which case the thermal connector allows a weak thermal connection initially to the cold body for precooling following which the connection to the cold body is strengthened so the sample cools to the desired final temperature.

[0011] Depending upon the temperature at which the working region is to be exposed, more than one heat radiation shield could be provided within the vacuum chamber. One or more of these could therefore be used further to precool the sample.

[0012] For example, in the preferred embodiment, the apparatus further comprises a second heat radiation shield located inside the first radiation shield and surrounding the working region, the cryogen free cooling system having a second cooling stage, colder than the first cooling stage, coupled to the second heat radiation shield, the second radiation shield having an aperture aligned with the apertures of the first heat radiation shield and vacuum chamber wall so as to allow the sample holding device to pass therethrough, whereby the sample holding device can be releasably coupled for heat conduction to the second heat radiation shield.

[0013] Where two or more heat shields are provided, it is not necessary for precooling of the sample to be carried out by connecting to each shield. For example, precooling could be carried out solely on the innermost (typically 4K) shield. If three or more shields are provided, one or more could be used for precooling.

[0014] Typically, the first heat radiation shield will be held at a temperature of between 45K and 90K while the second radiation shield (if provided) will be held at a temperature of less than 6K or even less than 4.2K.

[0015] The heat radiation shield apertures may be left open but in order to reduce heat transfer, preferably each aperture is closable by a respective closure system. An example of a suitable closure system comprises one or more flexible flaps, or hinged and sprung flaps.

[0016] In one embodiment, the sample loading apparatus comprises two elongate probes, each coupled to the sample holding device, but in other embodiments a single elongate probe could be used. In both cases, preferably the or each probe is rotatable about its axis relative to the sample holding device. Of course, more than two probes could be used.

[0017] The connector is conveniently formed by providing a screw thread at one end of the or each rod, the first connector cooperating with a screw thread on the first or second heat radiation shield to achieve thermal connection therebetween. Alternatively, the thermal connection can be achieved using a spring connection where the sample holding device is fitted with a or a plurality of thermally conductive springs which engage on an inner surface of the aperture of the radiation shield. That inner surface may be extended, for example by addition of a tube assembly or a thicker plate assembly to allow for engagement. The spring connectors could also be fixed on the heat or radiation shield and the sample holding device pushed on to them. Alternatively, the thermal connection could be via springs at the higher temperature shields and via screw contact at the lower temperature shields or any combination thereof. In another embodiment, the connector could be defined by cone or wedge-shaped mating parts to amplify the contact pressure from the mounting mechanism. This significantly improves performance.

[0018] In the case mentioned above where the connector initially provides a weak thermal connection, this could be by partially doing up the screws for precool and then fully doing them up once precooled (when screws are provided), or alternatively by initially pushing into spring contacts and then once precooled, tightening the clamp screws.

[0019] In a particularly preferred embodiment, the or each probe is releasably coupled to the sample holding device whereby a first operation of the probe(s) causes the sample holding device to be connected to a support at the working region, and a second operation enables the probe(s) to be released from the sample holding device and retracted. This enables the probe(s) to be removed from the vacuum chamber of the cryostat so as to reduce heat flow into the cryostat. Actuators to allow this could be provided on the probe or cold body.

[0020] The cryogen free cooling apparatus can be used for a variety of purposes such as DNP, NMR etc. and typically a magnet will be located within the cryostat surrounding the working region.

[0021] It should be understood that the sample loading apparatus just described, particularly the releasable nature of the sample holding device, is considered inventive in its own right and separate from the concept of precooling the sample as defined in the first and second aspects of the invention.

[0022] Some examples of apparatus and methods according to the invention will now be described with reference to the accompanying drawings, in which:-

Figure 1 shows a cutaway, part sectional view of a first embodiment of the sample loading apparatus;

Figure 2 shows a view of a first embodiment of the sample loading apparatus with the sample holding device retracted from the shields (for clarity the shields and electrical connectors are not shown);

Figure 3 shows a view similar to Figure 2 of the first embodiment but with the sample holding device connected to a cold plate in the cryostat (for clarity the shields are not shown);

Figure 4 shows a detail sectional view of the first embodiment of the sample loading apparatus;

Figure 5 shows a detail sectional view of the first embodiment showing a possible mechanism for closing the port in the shield when the sample is loaded (for clarity the shields and electrical connectors are not shown);

Figure 6 shows a cutaway view of a second embodiment of the sample loading apparatus; and

Figure 7 shows a cutaway view of the sample holding device and mating part of a second embodiment of the sample loading apparatus.

Detailed Descriptions of Specific Embodiments

[0023] A first embodiment of the current invention is shown in more detail in Figures 1 to 5. In Figure 1, a sample 1 is mounted on a sample carrier or sample loading device 2 supported on thermally conductive rods of two rod or probe assemblies 3. The sample carrier 2 has space for a number of electrical and/or optical connectors (not shown) to allow connection to connectors on the primary cold body in the cryostat. This allows multiple push fit connectors to be used which gives high flexibility and the wiring to go through the cryostat rather than down the probe tube, which has significant thermal benefits. The ends of the two rod assemblies 3 are free to rotate within the carrier. A tube and flange assembly forms a vacuum vessel 6 surrounding the rod assemblies 3 and which is open at one end, this end being sealed against the bottom of a gate valve 5 when assembled to a cryostat 50. At the opposite end of the vacuum vessel 6, the rod assemblies pass through a pair of o-ring seals 7. There is a separate vacuum space 9 and port 8A between these seals to allow any air leaking through the first seal, when the rod assemblies are moved, to be pumped away through a valve 8.

[0024] The cryostat 50 comprises an outer vacuum vessel 4 which is closed except for a port 52 covered by a large diameter gate valve 5. Within the vacuum vessel 4 is located a first radiation shield 54 having an aperture 56 aligned with the aperture 52 of the vacuum chamber, and within the first radiation shield 54 is located a second radiation shield 10 having an aperture 58 aligned with the apertures 52,56. The radiation shields 10,54 surround a working region 20 at which is located a cold mounting body 15.

[0025] The shields 10,54 are cooled by a conventional mechanical cooler such as a GM cooler, Stirling cooler, or pulse tube device. This is not shown in the drawings for reasons of clarity. A first stage of the mechanical cooler is thermally coupled to the shield 54 and a second, colder stage to the shield 10. Typically, the first shield 54 is cooled to a temperature of about 77K and the second shield 10 to a temperature of 6K or less, for example about 4.2K. In some cases, the second shield is held at a temperature higher than 6K. Thus, each of the shields as well as the cold mounting body 15 held at the lowest temperature can be considered as "cold bodies".

[0026] As can be seen in Figure 2, the aperture 56 of the shield 54 is defined by a plate 12 with a cut-out 17. Similarly, the aperture 58 of the shield 10 is defined by another plate 12 and cut-out 17.

[0027] Optionally, the apertures 56,58 can be closed by a suitable closure mechanism. Figure 5 shows a close up cross-sectional view of one possible embodiment of such a mechanism. A or a plurality of flaps 25 are connected to the radiation shield 10 via a sprung hinge arrangement 26. When the rod assembly 3 passes through the flap assembly, the flap or plurality thereof 25 open. The flap or plurality thereof 25 may optionally be shaped or fitted with guide mechanisms to prevent the sample carrier, baffles or rod assemblies from catching on the flaps as the rod assembly and/or carrier is retracted.

[0028] In operation, a sample 1 is loaded on to the sample carrier 2 and electrical or optical connections are made. The sample carrier 2 is then mounted on the end of the rod assemblies 3. The rod assemblies 3 are then retracted through the sliding o-ring seals 7 until the sample carrier is fully within the vacuum vessel 6. The vacuum vessel 6 is then attached to the gate valve 5 and air is pumped out of the vacuum vessel 6 through ports 8A,8B and valves 8. When a vacuum is established on both sides of the gate valve 5, the gate valve is opened. The rod assemblies 3 are then pushed to move the sample carrier through the gate valve and to the first pre-cool position.

[0029] Figure 2 shows the sample carrier 2 approaching the plate 12 of the shield 54 to thermally connect the sample carrier to a radiation shield pre-cool position defining a first cold body. The rod assemblies 3 have a key 22 (Figure 4) on the end which, when engaged, turns a screw thread 18. The screw threads 18 are aligned with mating screw threads 19 on the plate 12 allowing the sample carrier 2 to be screwed to the plate 12 on the radiation shield 54, thereby making thermal contact. An optional thermometer (not shown) is provided on the sample carrier or rod assembly to allow the temperature of the sample carrier to be monitored during cool down. When the sample carrier 2 is sufficiently cold, the rod assemblies 3 are again rotated to separate the two screw threads. The entire rod and carrier assembly is then rotated by means of a rotating seal on the vacuum vessel 6 or gate valve 5, to allow the carrier 2 to pass through the cut-out 17. The carrier is then optionally connected in a similar manner to a or a plurality of optional additional radiation shields, such as the shield 10 (forming additional cold bodies).

[0030] Once the sample carrier is suitably pre-cooled, the rod assemblies 3 are pushed to their final position to allow connection of the sample carrier 2 to the cold body 15 which could by way of example be connected to the mixing chamber of a dilution refrigerator or a sample plate of a cryostat. Figure 3 shows the sample carrier 2 contacting the cold plate 15. The screw threads 18 are engaged in mating screw threads (not shown) on the cold plate 15. During the thermal connection between the sample carrier 2 and the cold body 5, a number of optional push fit electrical and optional optical connections can be made between the sample carrier 2 and the cold body 15. These connectors are not shown on this diagram. In this view, two baffle assemblies 14 are also visible. These baffle assemblies are free to slide on the rod assemblies 3 and are pushed or pulled towards the sample carrier by spring assemblies 21. For clarity the baffle assemblies 14 are shown here in a retracted position, in reality they will be forced by the spring assemblies to contact the plates on the radiation shield, thereby closing the cut-outs 17 and making thermal contact. The baffle assemblies are also optionally connected to the rod assemblies using sliding thermal connections such as thermally conductive spring assemblies, thus allowing the heat passing down the rods from room temperature to be intercepted.

[0031] Figure 4 shows a close up cross sectional view of the sample carrier and rod assemblies. On the end of each rod assembly 3 there is the key 22 that inserts into a matching connection on the screw thread 18. On the key and rod assembly, there is a screw thread 23 and on the sample carrier there is a matching screw thread 24. This arrangement means that if the rod assemblies are retracted, the screw threads 23,24 will clash and the sample carrier will therefore also be retracted. Once the sample carrier is connected to the cold body 15 by means of the screw threads 18 the rod assemblies can then be partially retracted to remove the key from the back of the screw thread 18 and reduce heat flow to the sample. However, this is not essential and the sample could remain connected to the probe. When the threads 23,24 clash, the rod assembly can then be rotated to allow the screw threads to pass through each other and then either be partially retracted from the cryostat, leaving the baffles in contact with the radiation shields, or be fully retracted from the cryostat in order to further reduce heat load.

[0032] If the rod assemblies are fully retracted from the cryostat, the optional mechanism 11 can be fitted to close the cut outs in the radiation shields.

[0033] A second embodiment of the current invention is shown in Figure 6. In this embodiment, a single rod assembly 3 is used with a single large diameter screw thread 18. On the end of the rod assembly 3 there is an adapter 27 which connects the rod assembly to the sample carrier assembly 2. On the adapter there are a or a plurality of protrusions 28 that engage in slots or recesses 29 formed on the means 12 to allow the carrier to be thermally connected to the radiation shields. The sample is loaded into the carrier and entered through the gate valve 5 as per the first embodiment. The rod assembly is rotated to engage the protrusions 28 in the slots or recesses 28 and the rod assembly is then pushed towards the cryostat until the protrusions 28 meet an obstruction 30. Thermal connection is then optionally made through the protrusions or through optional spring contacts 31. The slot and obstruction are optional and serve to prevent the sample carrier from being accidentally pushed past the radiation shield prior to pre-cooling.

[0034] When the sample is cooled adequately, the sample rod is optionally retracted slightly and rotated to allow the protrusions 28 to move past the obstruction 30. The rod assembly can then be further inserted to allow it to be thermally connected to the next radiation shield if so required. When the sample rod is inserted through the shield, the optional baffles 13 fitted with optional spring thermal contacts 14 engage in the assembly 12 so as to both close the port in the radiation shield and optionally to make thermal contact between the radiation shield and the rod assembly to intercept heat. A similar optional process for pre-cooling on subsequent radiation shield(s) can then be included before moving the sample to the cold body.

[0035] Figure 7 shows a cross sectional view of the sample carrier assembly of the second embodiment engaged on the cold body. The sample carrier 2 is enclosed in a tube 32 with a screw thread 18 on one end. At the opposite end of the tube a means 33 of connecting the tube to the adapter on the end of the rod assembly is provided. This allows the tube to be inserted and retracted and to be rotated by the rod assembly. The sample carrier is free to rotate inside the tube and is thermally connected to the adapter at the end of the rod assembly using a spring thermal contact 34. As the tube and carrier assembly is pushed on to the mating part attached to the cold body, a keyway rotationally aligns the sample carrier to the mating part, ensuring that the optional connectors 35 align. The rod assembly is then rotated to pull the sample carrier on to the mating part, making the thermal contact and optional electrical and optical connections. The rod assembly can then be retracted from the cryostat, disconnecting at the means of connecting the tube to the adapter on the end of the rod assembly. Optional baffles can be fitted to close the ports in the radiation shields if the rod assembly is to be completely removed. Removal of the sample is essentially the reverse of the insertion process, with the exception that it is not usually necessary to leave the sample carrier at the radiation shields to warm up when retracting the sample.

Alternative embodiments:

[0036] In the first alternative embodiment, it is possible to change the mechanism for connection to the radiation shields from being a screw connection to being a spring connection where the sample carrier is fitted with a or a plurality of thermally conductive springs which engage on an inner surface of the cut-out on the radiation shield. That inner surface may be extended, for example by addition of a tube assembly or a thicker plate assembly to allow for engagement. Alternatively, the thermal connection could be via springs at the higher temperature shields and via screw contact at the lower temperature shields or any combination thereof. Cone or wedge-shaped mating parts on either side of the releasable coupling could be used to amplify the contact pressure from the mounting mechanism. Pneumatic or piezo or other forms of releasable contact could also be used.

[0037] In all embodiments, the connection to the or each cold body can optionally be via thermally conductive spring contacts rather than screw connection.

[0038] In all embodiments, the connection to the radiation shields can optionally be via thermally conductive spring contacts or screw contacts.

[0039] In all embodiments, where it is specified that a thermal connection is or could be made to a radiation shield or shield, this thermal connection could alternatively be made to any other suitable cold surface.

[0040] In all embodiments, as an alternative to precooling at progressively lower temperatures at radiation shields, the sample and carrier may optionally be precooled by making a weaker thermal contact to a colder temperature body, such as the coldest body. The relative warming of the cryostat and cooling of the sample and carrier can be controlled by design of the thermal contact.

[0041] Wherever thermally conductive spring contacts are used, these can be made from a single material, such as Berillium Copper, or may be made from a laminate or composite of different materials to provide both a good spring force and a high thermal conductivity. This could for example include Berillium Copper or steel to provide the spring force with copper, silver and or gold to enhance the thermal conductivity. Dissimilar materials are preferred so as to reduce eddy currents and quench forces when used with a magnet. Examples of dissimilar materials could be copper for high thermal conductivity and stainless steel for high strength and lower electrical conductivity to reduce induced eddy currents. Other possibilities could include titanium and copper or brass and copper or alumium alloy and copper. Generically, it is one material of high thermal conductivity and one of high strength and higher resistance. The second material could also be a plastic or a composite.

[0042] In all embodiments, an additional port or plurality thereof can be added to the second vacuum vessel to allow the sample and optionally the sample carrier to be removed without removal of the second vacuum vessel from the main vacuum vessel.

[0043] In the second embodiment, it is possible to change the connection to the radiation shields to a screw thread on the outside of the rotating tube assembly. It is also possible to change the screw thread connection to the cold body to be an external thread, meaning the same thread can be used to connect to the radiation shields for pre-cooling and then to the cold body. The tube assembly with the thread may optionally have a split in it to allow the diameter to change to compensate for thermal expansion and contraction.

[0044] Although not shown, a superconducting magnet could be located in the cryostat 50 as is known conventionally for dynamic nuclear polarisation and nuclear magnetic resonance and other cryogenic magnetic field applications.

[0045] In the examples described above, the rods form actuators for connecting and disconnecting to the cold bodies and are demountable from the cryostat. In alternative examples, the rods (or other actuators) could form part of the cryostat and the sample carrier could be carried on a probe independent of the rods (or other actuators), the rods (or other actuators) being manipulated to engage the screw threads (or other connection mechanism) as before.

[0046] Further exemplary embodiments of the present disclosure are set out in the following numbered clauses:

Numbered clause 1. A cryogen free cooling apparatus comprising at least one heat radiation shield surrounding a working region and located in a vacuum chamber; a cryofree cooling system having a cooling stage coupled to the heat radiation shield; aligned apertures in the heat radiation shield and vacuum chamber wall; sample loading apparatus having a sample holding device attached to one or more elongate probes for inserting the sample holding device through the aligned apertures to the working region; and a thermal connector, whereby the sample holding device can be releasably coupled for heat conduction via said connector to a cold body or cold bodies within the vacuum chamber so as to pre-cool a sample on or in the sample holding device.

Numbered clause 2. Apparatus according to clause 1, wherein the or one cold body is formed by a surface linked to a cold plate coupled to a cooling stage of the cooling system.

Numbered clause 3. Apparatus according to clause 1 or clause 2, wherein the or one cold body is formed by a heat radiation shield, whereby the first connector can be releasably coupled for heat conduction to the heat radiation shield.

Numbered clause 4. Apparatus according to clause 3, further comprising a second heat radiation shield located inside the first radiation shield and surrounding the working region, the cryogen free cooling system having a second cooling stage, colder than the first cooling stage, coupled to the second heat radiation shield, the second radiation shield having an aperture aligned with the apertures of the first heat radiation shield and vacuum chamber wall so as to allow the sample holding device to pass therethrough, whereby the sample holding device can be releasably coupled for heat conduction to the second heat radiation shield acting as the or a cold body.

Numbered clause 5. Apparatus according to clause 4, wherein the second heat radiation shield is held at a temperature of less than 6K.

Numbered clause 6. Apparatus according to any of the preceding clauses, wherein the first heat radiation shield is held at a temperature of between 45K and 90K.

Numbered clause 7. Apparatus according to any of the preceding clauses, wherein the cold body is the coldest body within the cooling apparatus, the thermal connector enabling a weak thermal connection to be achieved with the sample holding device and subsequently a stronger thermal connection allowing a greater rate of heat flow.

Numbered clause 8. Apparatus according to any of the preceding clauses, wherein the vacuum chamber aperture includes a closure system such as a vacuum valve.

Numbered clause 9. Apparatus according to any of the preceding clauses, wherein the or each heat radiation shield aperture is closable by a respective closure system.

Numbered clause 10. Apparatus according to clause 9, wherein the closure system for the aperture of the or each heat radiation shield comprises one or more flexible flaps or hinged and sprung flaps.

Numbered clause 11. Apparatus according to any of the preceding clauses, wherein the sample loading apparatus comprises two or more elongate probes, each coupled to the sample holding device.

Numbered clause 12. Apparatus according to clause 11, wherein the or each probe is rotatable about its axis relative to the sample holding device.

Numbered clause 13. Apparatus according to clause 11 or clause 12, wherein the or each probe is screw threaded at one end to define the thermal connector, the connector cooperating with a screw thread on the or one of the cold bodies to achieve a thermal connection therebetween.

Numbered clause 14. Apparatus according to any of clauses 1 to 11, wherein one or more of the thermal connectors comprises one or more thermally conductive springs fitted to make thermal contact between the or one cold body and the sample holding device.

Numbered clause 15. Apparatus according to clause 14, wherein the conductive springs comprise composite material with high thermal conductivity and high spring force.

Numbered clause 16. Apparatus according to any of the preceding clauses, wherein the sample loading apparatus is rotatable relative to the vacuum chamber and heat shield(s) so as selectively to align with the or each thermal connector or with the respective aperture so as to allow the sample holding device to be passed therethrough.

Numbered clause 17. Apparatus according to any of the preceding clauses, wherein the or each probe is releasably coupled to the sample holding device whereby a first operation of the probe(s) causes the sample holding device to be connected to a support at the working region, and a second, subsequent operation enables the probe(s) to be released from the sample holding device and retracted.

Numbered clause 18. Apparatus according to any of the preceding clauses, wherein the sample loading apparatus further includes a vacuum vessel in which the sample holding device and elongate probe or probes are movably mounted, the vacuum vessel being connectable to the aperture of the vacuum chamber wall.

Numbered clause 19. Apparatus according to any of the preceding clauses, wherein the cryogen free cooling system comprises a mechanical cooler such as a GM cooler, Stirling cooler or pulse tube device.

Numbered clause 20. Apparatus according to any of the preceding clauses wherein the sample holder includes one or more electrical connectors to allow electrical and thermal connections to be made to a cold body in the working region.

Numbered clause 21. Apparatus according to any of the preceding clauses wherein the sample holder includes one or more optical connectors such as fiber optic connectors to allow electrical and thermal connections to be made to a cold body in the working region.

Numbered clause 22. A method of loading a sample into the working region of cryogen free cooling apparatus according to at least clause 18, the method comprising

placing a sample in or on the sample holding device;
securing the vacuum vessel of the sample loading apparatus to the vacuum chamber and aligned with the aperture of the vacuum chamber;
evacuating the vacuum vessel;
opening the aperture of the vacuum chamber and operating the or each elongate probe to insert the sample holding device through the opened aperture so that the sample holding device is thermally coupled to a cold body;
allowing the sample in or on the sample holding device to be cooled as a result of heat conduction to the cold body; and either

a) disconnecting the sample holding device from the cold body; and operating the or each elongate probe to insert the sample holding device into the working region, or
b) strengthening the thermal connection to the cold body to increase heat flow away

from the sample and further cool the sample.

Numbered clause 23. A method according to clause 22, when dependent on clause 4, wherein prior to reaching the working region, the sample holding device is thermally coupled to the second heat radiation shield, cooled by allowing heat to flow to the second radiation shield, disconnected from the second radiation shield, and the sample holding device is then inserted into the working region.

Numbered clause 24. A method according to clause 22 or clause 23, further comprising detaching the sample holding device from the or each elongate probe following insertion into the working region, and withdrawing the or each elongate probe and subsequently closing the closure system of the aperture of the vacuum chamber.

Claims

1. A cryogen free cooling apparatus comprising:

a vacuum chamber (4);
a first heat radiation shield (54) surrounding a working region (20) and located in the vacuum chamber;
a cryofree cooling system having a first cooling stage coupled to the first heat radiation shield;
a cold body within the vacuum chamber, wherein the cold body is formed by a surface linked to a cold plate coupled to a cooling stage of the cooling system;
aligned apertures (52, 56) in the first heat radiation shield and vacuum chamber wall;
sample loading apparatus having one or more elongate probes (3) and a sample holding device (2) attached to the one or more elongate probes for inserting the sample holding device through the aligned apertures (52, 56) to the working region;
one or more thermal connectors, whereby the sample holding device is releasably connected for heat conduction via said thermal connector(s) to the cold body so as to pre-cool a sample (1) on or in the sample holding device (2) before the sample holding device is inserted into the working region (20); and
a support (15) located at the working region;
characterised in that the sample holding device (2) includes one or more electrical connectors to allow electrical and thermal connections to be made to the support (15) in the working region (20).

2. Apparatus according to claim 1, wherein the cold body is formed by the first heat radiation shield (54).

3. Apparatus according to claims 2, further comprising a second heat radiation shield (10) located inside the first radiation shield (54) and surrounding the working region (20), the cryogen free cooling system having a second cooling stage, colder than the first cooling stage, coupled to the second heat radiation shield, the second radiation shield having an aperture (58) aligned with the apertures (52, 56) of the first heat radiation shield and vacuum chamber wall so as to allow the sample holding device (2) to pass therethrough, whereby the sample holding device can be releasably coupled for heat conduction to the second heat radiation shield.

4. Apparatus according to any of claims 1 to 3, wherein the support (15) is connected to the mixing chamber of a dilution refrigerator.

5. Apparatus according to any of the preceding claims, wherein the aligned aperture (52) in the vacuum chamber wall includes a closure system (5), such as a vacuum valve.

6. Apparatus according to any of the preceding claims, wherein one or more of the thermal connector(s) comprises one or more thermally conductive springs (31) fitted to make thermal contact between the cold body and the sample holding device.

7. Apparatus according to claims 2 and 6, wherein the sample holding device is fitted with the thermally conductive spring(s) to engage on an inner surface of the aperture of the first radiation shield.

8. Apparatus according to claims 2 and 6, wherein said thermally conductive spring(s) are fixed on the first heat radiation shield.

9. Apparatus according to claim 3, wherein the sample loading apparatus is rotatable relative to the vacuum chamber (4) and the first and second heat shields (10, 54) so as selectively to align with the or each thermal connector or with the respective aperture so as to allow the sample holding device to be passed therethrough.

10. Apparatus according to any of the preceding claims, wherein the probe or each of the elongate probes (3) is releasably coupled to the sample holding device for releasing and retracting the probe(s) from the sample holding device when the sample holding device is connected to the support.

11. Apparatus according to any of the preceding claims, wherein the sample loading apparatus further includes a vacuum vessel (6) in which the sample holding device (2) and elongate probe or probes (3) are movably mounted, the vacuum vessel being connectable to the aperture (52) of the vacuum chamber wall.

12. A method of loading a sample into the working region of a cryogen free cooling apparatus according to claims 5 and 11, the method comprising

placing a sample (1) in or on the sample holding device (2);
securing the vacuum vessel (6) of the sample loading apparatus to the vacuum chamber (4) and aligned with the aperture (52) of the vacuum chamber;
evacuating the vacuum vessel;
opening the aperture of the vacuum chamber and operating the or each elongate probe (3) to insert the sample holding device through the opened aperture so that the sample holding device is thermally coupled to the cold body;
allowing the sample in or on the sample holding device to be cooled as a result of heat conduction to the cold body;
disconnecting the sample holding device from the cold body; and operating the or each elongate probe to insert the sample holding device into the working region.

13. A cryogen free cooling apparatus comprising:

a vacuum chamber (4);
a heat radiation shield (54) surrounding a working region (20) and located in the vacuum chamber;
a cryofree cooling system having a cooling stage coupled to the heat radiation shield;
a cold body within the vacuum chamber, wherein the cold body is formed by a surface linked to a cold plate coupled to a cooling stage of the cooling system;
aligned apertures (52, 56) in the heat radiation shield and vacuum chamber wall;
sample loading apparatus having one or more elongate probes (3) and a sample holding device (2) attached to the one or more elongate probes for inserting the sample holding device through the aligned apertures (52, 56) to the working region (20); and
a support (15) located at the working region (20), whereby the sample holding device (2) is configured to be connected to the support by operation of the probe(s) (3) and whereby the elongate probe or each of the elongate probes is releasably coupled to the sample holding device for releasing and retracting the probe(s) from the sample holding device when the sample holding device is connected to the support;

characterised by one or more thermal connectors, whereby the sample holding device (2) is releasably connected for heat conduction via said connector(s) to the cold body so as to precool a sample on or in the sample holding device before the sample holding device is connected to the support.

14. A method of loading a sample (1) into the working region (20) of cryogen free cooling apparatus, the cryogen free cooling apparatus comprising:

a vacuum chamber (4);
a heat radiation shield (54) surrounding a working region (20) and located in the vacuum chamber (4);
a cryofree cooling system having a cooling stage coupled to the heat radiation shield (54);
a cold body within the vacuum chamber, wherein the cold body is formed by a surface linked to a cold plate coupled to a cooling stage of the cooling system;
a support (15) located at the working region (20);
aligned apertures (52, 56) in the heat radiation shield (54) and vacuum chamber wall, wherein the aligned aperture (52) in the vacuum chamber wall includes a closure system (5) such as a vacuum valve;
sample loading apparatus having one or more elongate probes (3) and a sample holding device (2) attached to the one or more elongate probes, the one or more elongate probes for inserting the sample holding device through the aligned apertures (52, 56) to the working region (20), wherein the sample loading apparatus further includes a vacuum vessel (6) in which the sample holding device (2) and elongate probe or probes (3) are movably mounted, the vacuum vessel being connectable to the aperture (52) of the vacuum chamber wall; and
one or more thermal connectors, wherein the sample holding device (2) is releasably connected for heat conduction via said connector(s) to the cold body (12) so as to pre-cool a sample (1) on or in the sample holding device before the sample holding device is connected to the support (15);

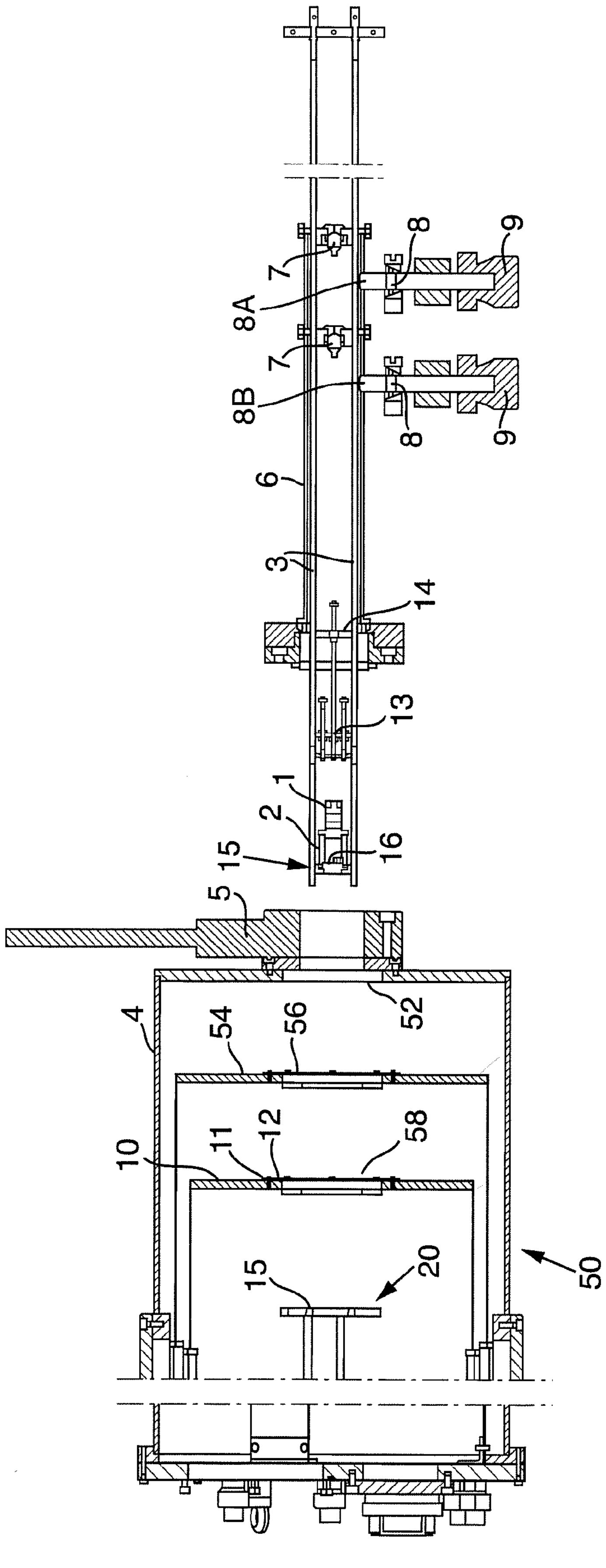
the method comprising:

placing a sample (1) in or on the sample holding device (2);
securing the vacuum vessel (6) of the sample loading apparatus to the vacuum chamber (4) and aligned with the aperture (52) of the vacuum chamber;
evacuating the vacuum vessel (6);
opening the aperture (52) of the vacuum cham-

ber (4) and operating the or each elongate probe (3) to insert the sample holding device (2) through the opened aperture so that the sample holding device is thermally coupled to the cold body;
allowing the sample (1) in or on the sample holding device (2) to be cooled as a result of heat conduction to the cold body;
disconnecting the sample holding device (2) from the cold body;
operating the or each elongate probe (3) to insert the sample holding device (2) into the working region (20);
connecting the sample holding device (2) to the support (15) via the connector(s); and
releasing and retracting the elongate probe or each of the elongate probes (3) from the sample holding device when the sample holding device is connected to the support.

15. A method according to claim 14, wherein releasing and retracting the elongate probe or each of the elongate probes (3) comprises partially retracting the elongate probe or each of the elongate probes from the sample holding device when the sample holding device is connected to the support (15).

Fig.1.



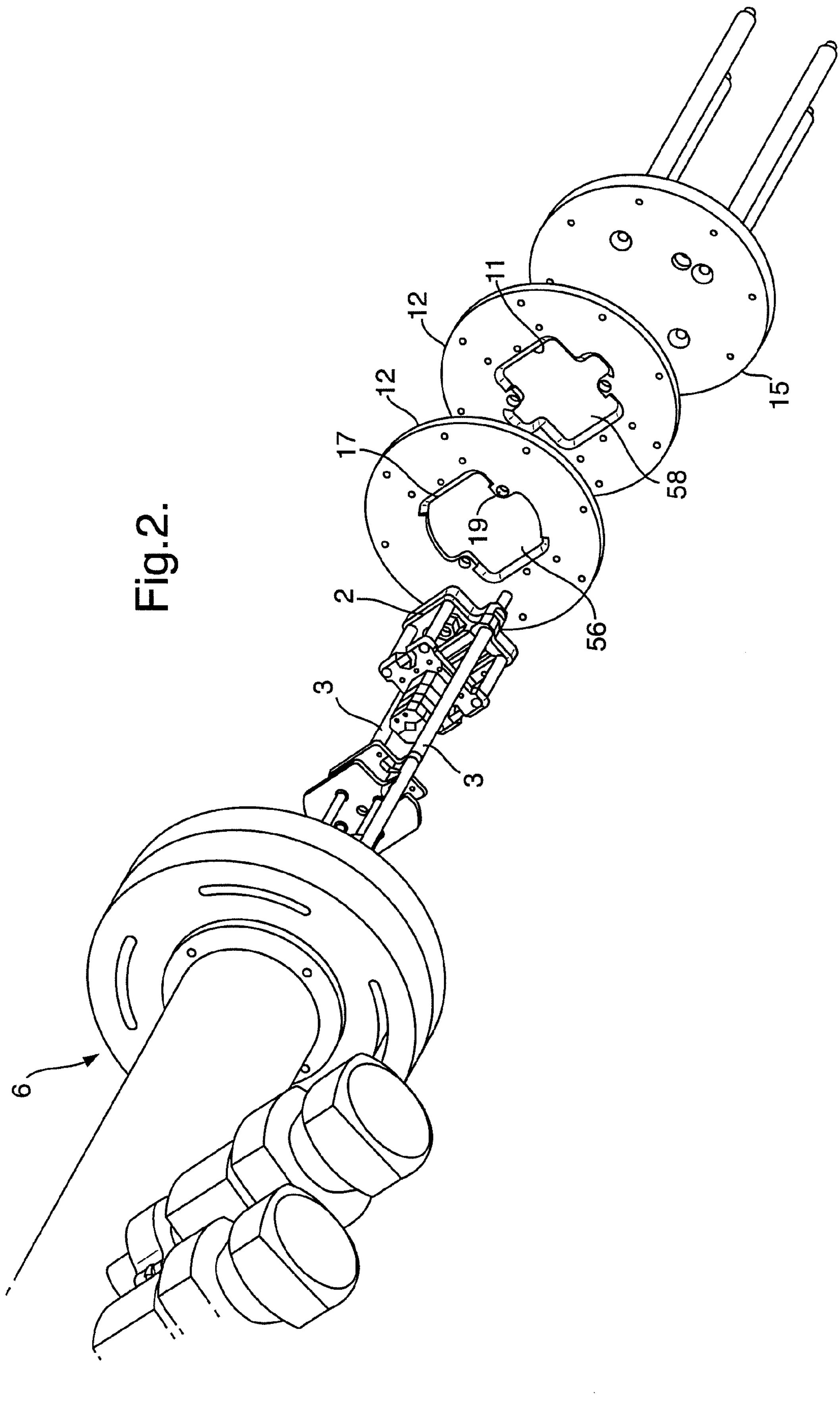


Fig.2.

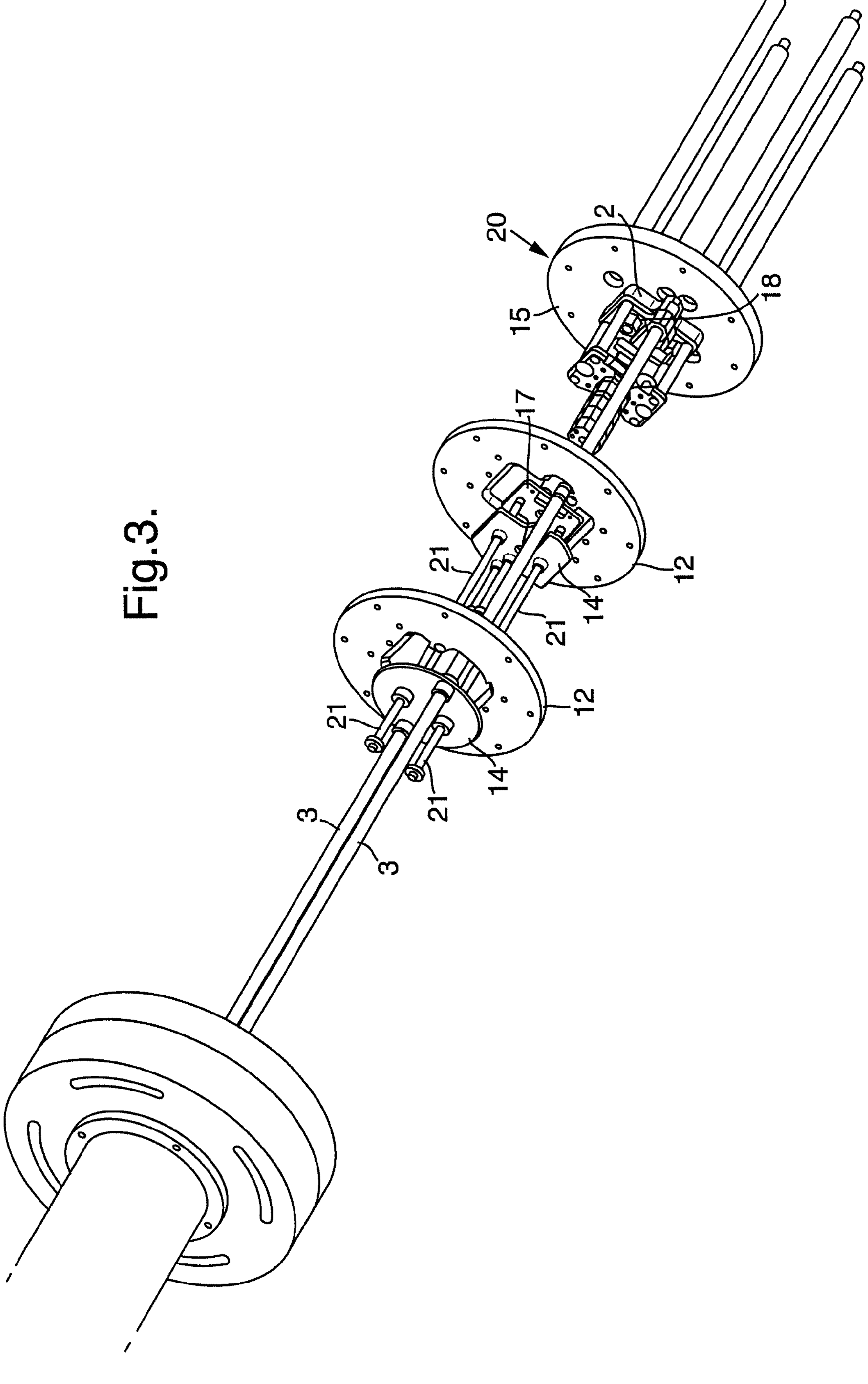


Fig.3.

Fig.4.

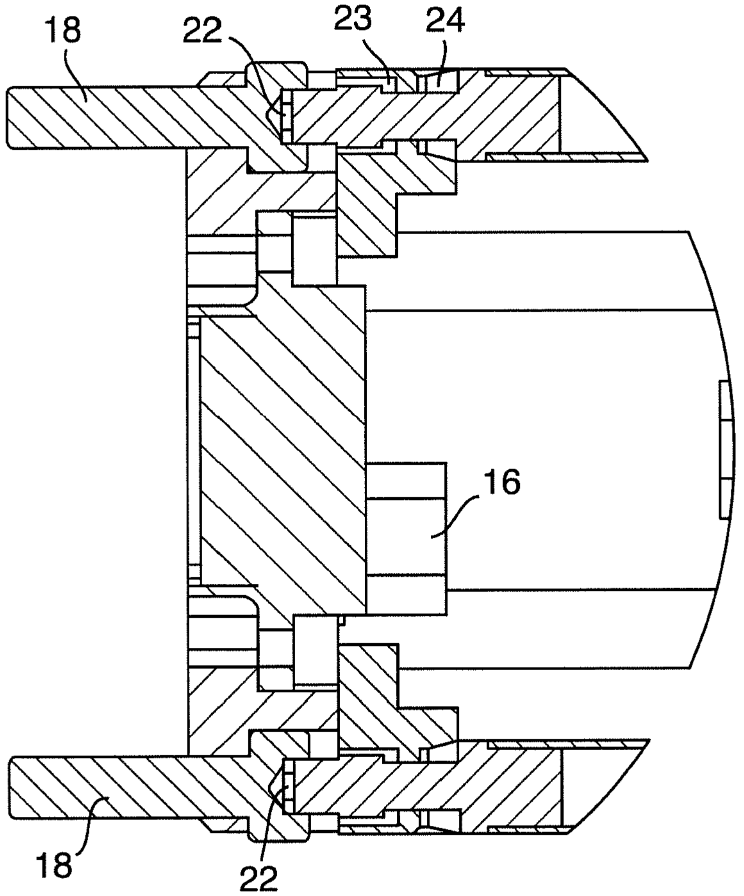


Fig.5.

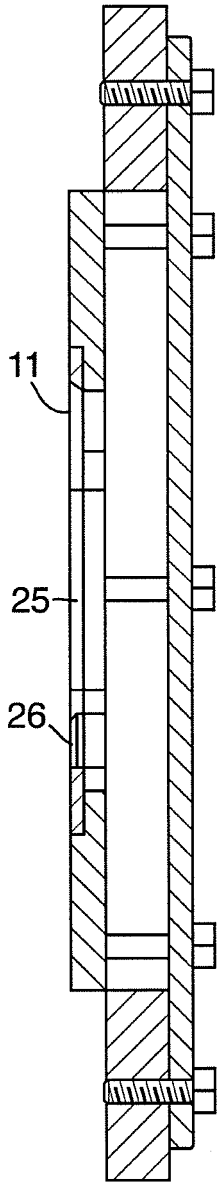


Fig.6.

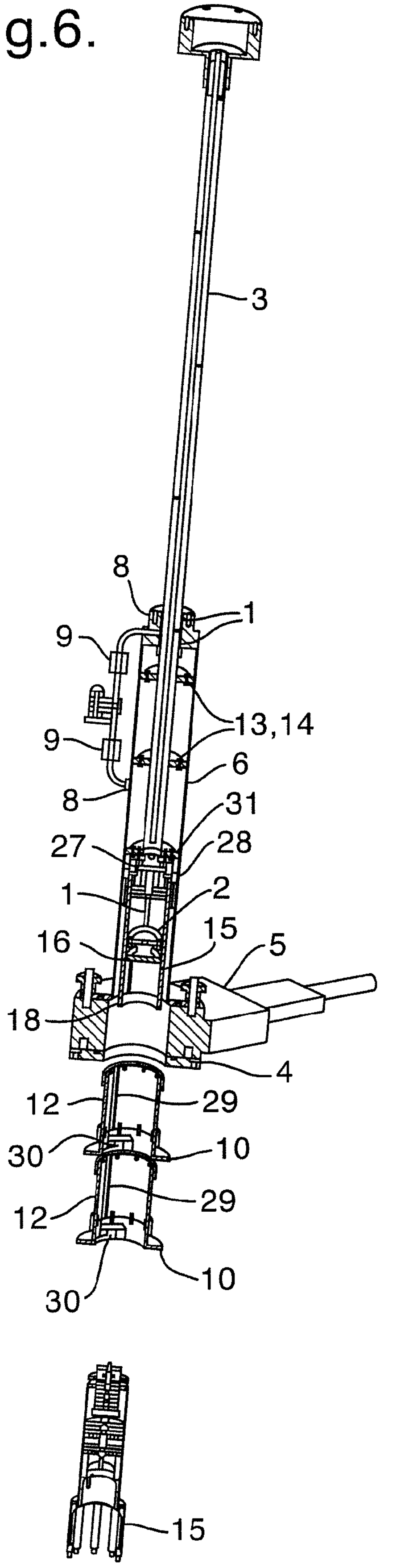
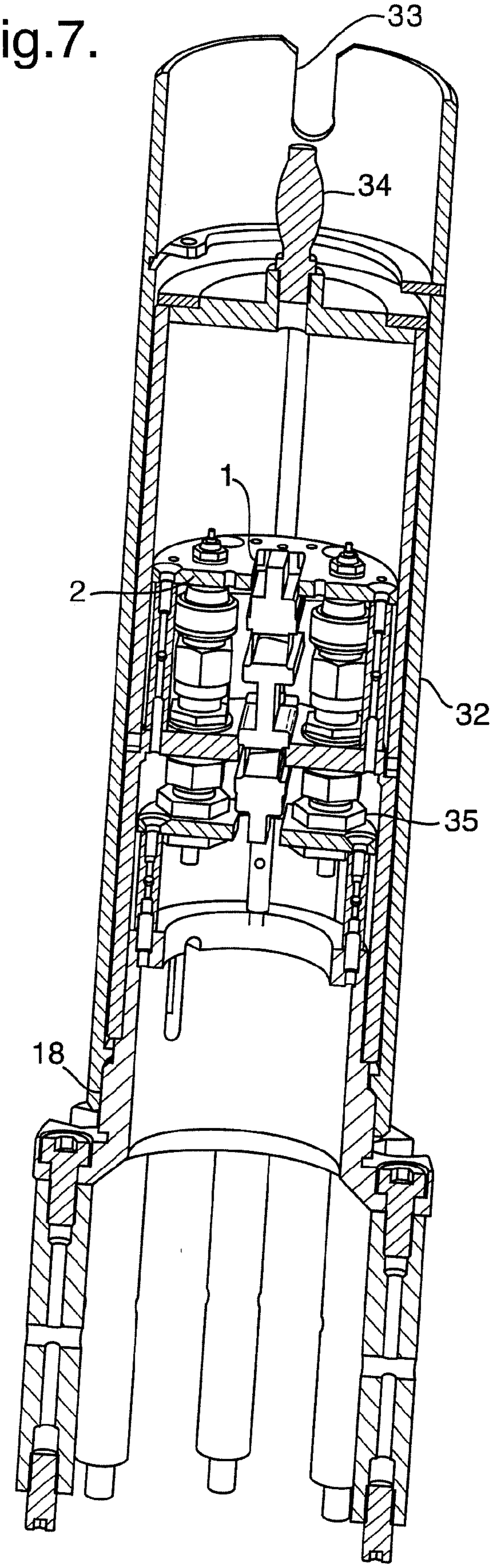


Fig.7.



REFERENCES CITED IN THE DESCRIPTION

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