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(54) CRYOGEN FREE COOLING APPARATUS AND METHOD

KRYOGENFREIE KÜHLVORRICHTUNG UND VERFAHREN

APPAREIL ET PROCÉDÉ DE REFROIDISSEMENT SANS CRYOGÈNE

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(56) References cited:
WO-A2-2009/000629
JP-A- 2008 014 878
US-A- 5 237 825
US-A- 5 611 207

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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] JP 2008-14878 A discloses a cryogen free cooling apparatus according to the preamble of claim 1.

[0007] Aspects of the invention are defined by the appended claims.

[0008] 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.

[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 pre-cool 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.

[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 pre-cool 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] According to one aspect of the invention, 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 cold mounting body at the working region. 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, scientific cryogenic research, quantum computing, experimental analysis, material characterisation, device characterisation, detector cooling, device cooling, DNP, NMR or any other application where cooling of matter to cryogenic temperatures is required and in many cases a magnet may be located within the cryostat surrounding the working region.

[0021] 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

[0022] 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 optionally for 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.

[0023] 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 chamber 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.

[0024] 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".

[0025] 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.

[0026] 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.

[0027] 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.

[0028] 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).

[0029] 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.

[0030] 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.

[0031] 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.

[0032] 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.

[0033] 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.

[0034] 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:

[0035] 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.

[0036] In all embodiments, the connection to the or each cold body can optionally be via thermally conductive spring contacts rather than screw connection.

[0037] In all embodiments, the connection to the radiation shields can optionally be via thermally conductive spring contacts or screw contacts.

[0038] 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.

[0039] 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.

[0040] 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.

[0041] 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.

[0042] 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.

[0043] 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.

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 (4);
a cryofree cooling system, for example a mechanical cooler such as a GM cooler, Stirling cooler or pulse tube device, having a cooling stage coupled to the first heat radiation shield (54);
a cold body (12) formed by a surface linked to a cold plate coupled to a cooling stage of the cooling system;
a cold mounting body (15) located at the working region (20) and held at a lower temperature than the cold body (12);
aligned apertures (52, 56) in the first heat radiation shield (54) 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, 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 thermal connector (18), whereby the sample holding device (2) is configured to be connected to the cold mounting body (15) via the connector, and whereby the elongate probe or each of the elongate probes (3) is relesably coupled to the sample holding device for releasing the sample holding device when the sample holding device is connected to the cold mounting body;
characterised in that the sample holding device (2) is releasably coupled for heat conduction via said connector (18) 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 cold mounting body (15).

2. Apparatus according to claim 1, wherein the cold body is formed by the first heat radiation shield (54), whereby the connector (18) can be releasably coupled for heat conduction to the first heat radiation shield.

3. Apparatus according to claim 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 claim 3, wherein the first heat radiation shield (54) is held at a temperature of between 45K and 90K, and the second heat radiation shield (10) is preferably held at a temperature of less than 6K.

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 the or each aligned aperture (56, 58) in the first heat heat radiation shield (54) and second radiation shield (10) is closable by a respective closure system (25), such as one or more flexible flaps or hinged and sprung flaps.

7. Apparatus according to any of the preceding claims, wherein the one or more elongate probes (3) comprise two or more elongate probes, each coupled to the sample holding device.

8. Apparatus according to claim 7, wherein each probe is rotatable about its axis relative to the sample holding device (2), and wherein the or each probe is preferably screw threaded (18) at one end to define the thermal connector, the connector cooperating with a screw thread (19) on cold body to achieve a thermal connection therebetween.

9. Apparatus according to any of claims 1 to 7, wherein the thermal connector comprises one or more thermally conductive springs, typically comprising composite material with high thermal conductivity and high spring force, fitted to make thermal contact between the cold body and the sample holding device (2).

10. 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 (18) or with the respective aperture (52, 56, 58) so as to allow the sample holding device (2) to be passed therethrough.

11. Apparatus according to any of the preceding claims wherein the sample holding device (2) includes one or more electrical connectors to allow electrical and thermal connections to be made to a cold body (15) in the working region (20).

12. Apparatus according to any of the preceding claims wherein the sample holding device (2) includes one or more optical connectors such as fiber optic connectors to allow electrical and thermal connections to be made to a cold body (15) in the working region (20).

13. 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.

14. A method of loading a sample (1) into the working region (20) of cryogen free cooling apparatus according claims 5 and 13, 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 chamber (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; and
operating the or each elongate probe (3) to insert the sample holding device (2) into the working region (20).

15. A method according to claim 14, wherein the apparatus further comprises 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 of the first heat radiation shield and vacuum chamber wall (52, 54) 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, and 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.

Patentansprüche

1. Kryogenfreie Kühlvorrichtung, umfassend:

Eine Vakuumkammer (4);
ein erstes Wärmestrahlungsschild (54), das einen Arbeitsbereich (20) umgibt und in der Vakuumkammern (4) positioniert ist;
ein kryogenfreies Kühlsystem, zum Beispiel ein mechanischer wie ein GM-Kühler, Stirling-Kühler oder ein Pulsröhrengerät, mit einer Kühlstufe, die an das erste Wärmestrahlungsschild (54) gekoppelt ist;
einen kalten Körper (12), der von einer Oberfläche gebildet ist, die mit einer kalten Platte verbunden ist, die an eine Kühlstufe des Kühlsystems gekoppelt ist;
einen kalten Montagekörper (15), der im Arbeitsbereich (20) positioniert ist und auf einer niedrigeren Temperatur als der kalte Körper (12) gehalten wird;
ausgerichtete Öffnungen (52, 56) im ersten Wärmestrahlungsschild (54) und der Vakuumkammerwand;
Probenladevorrichtung mit einer oder mehreren Sonden (3) und ein Probenhaltegerät (2), das an die eine oder mehrere der länglichen Sonden angebracht ist, die eine oder mehrere längliche Sonden zum Einführen des Haltegeräts durch die ausgerichteten Öffnungen (52, 56) in den Arbeitsbereich (20); und
einen thermischen Anschluss (18), wodurch das Probenhaltegerät (2) konfiguriert ist, über den Anschluss mit dem kalten Montagekörper (15) verbunden zu sein, und wobei die längliche Sonde oder jede der länglichen Sonden (3) lösbar an das Probenhaltegerät gekoppelt sind, um das Probehaltegerät freizugeben, wenn das Probenhaltegerät mit dem kalten Montagekörper verbunden ist;
dadurch gekennzeichnet, dass das Probenhaltegerät (2) für Wärmeleitung über den Anschluss (18) lösbar an den kalten Körper (12) gekoppelt ist, um eine Probe (1) auf oder im Probenhaltegerät vorzukühlen, bevor das Proben-

haltegerät an den kalten Montagekörper (15) angeschlossen wird.

2. Vorrichtung nach Anspruch 1, wobei der kalte Körper durch das erste Wärmestrahlungsschild (54) gebildet wird, wodurch der Anschluss (18) lösbar für Wärmeleitung an das erste Wärmestrahlungsschild gekoppelt werden kann.

3. Vorrichtung nach Anspruch 2, die ferner ein zweites Wärmestrahlungsschild (10) umfasst, das innerhalb des ersten Wärmeschilds (54) positioniert ist und den Arbeitsbereich (20) umgibt, wobei das kryogenfreie Kühlsystem eine zweite Kühlstufe aufweist, kälter als die erste Kühlstufe, die an das zweite Wärmestrahlungsschild gekoppelt ist, wobei das zweite Wärmeschild eine Öffnung (58) aufweist, die mit den Öffnungen (52, 56) des ersten Wärmestrahlungsschilds und der Vakuumkammerwand ausgerichtet ist, um dem Probenhaltegerät (2) zu erlauben dort hindurch zu gehen, wodurch das Probenhaltegerät für Wärmeleitung lösbar an das zweite Wärmestrahlungsschild gekoppelt werden kann.

4. Vorrichtung nach Anspruch 3, wobei das erste Wärmestrahlungsschild (54) auf einer Temperatur von zwischen 45K und 90K gehalten wird und das zweite Wärmestrahlungsschild (10) vorzugsweise auf einer Temperatur von weniger als 6K gehalten wird.

5. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die ausgerichtete Öffnung (52) in der Vakuumkammerwand ein Verschlusssystem (5) wie beispielsweise ein Vakuumventil einschließt.

6. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die oder jede ausgerichtete Öffnung (56, 58) im ersten Wärmestrahlungsschild (54) und zweiten Wärmestrahlungsschild (10) durch ein jeweiliges Verschlusssystem (25), wie beispielsweise eine oder mehrere flexible Klappen oder scharnierte oder gefederte Klappen, schließbar ist.

7. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die eine oder die mehreren länglichen Sonden (3) zwei oder mehrere längliche Sonden umfassen, wobei jede an das Probenhaltegerät gekoppelt ist.

8. Vorrichtung nach Anspruch 7, wobei jede Sonde um ihre Achse relativ zum Probenhaltegerät (2) drehbar ist, und wobei die oder jede Sonde vorzugsweise an einem Ende mit einem Schraubengewinde (18) versehen ist, um den thermischen Anschluss zu definieren, wobei der Anschluss mit einem Schraubengewinde (19) am kalten Körper kooperiert, um eine thermische Verbindung dazwischen zu erzielen.

9. Vorrichtung nach einem der Ansprüche 1 bis 7, wobei der thermische Anschluss eine oder mehrere thermisch leitfähige Federn umfasst, die typisch Verbundmaterial mit hoher thermischer Leitfähigkeit und hoher Federkraft umfassen, angebracht zur Herstellung von thermischem Kontakt zwischen dem kalten Körper und dem Probenhaltegerät (2).

10. Vorrichtung nach Anspruch 3, wobei die Probenladevorrichtung relativ zur Vakuumkammer (4) und den ersten und zweiten Wärmeschildern (10, 54) drehbar ist, um sich selektiv mit dem oder jedem thermischen Anschluss (18) oder mit der jeweiligen Öffnung (52, 56, 58) auszurichten, um dem Probenhaltegerät (2) dort hindurch geleitet zu werden.

11. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei das Probenhaltegerät (2) einen oder mehrere elektrische Anschlüsse einschließt, um elektrische und thermische Anschlüsse zu einem kalten Körper (15) im Arbeitsbereich (20) herzustellen zu können.

12. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei das Probenhaltegerät (2) einen oder mehrere optische Anschlüsse, wie beispielsweise Glasfaseranschlüsse, einschließt, um elektrische und thermische Anschlüsse zu einem kalten Körper (15) im Arbeitsbereich (20) herzustellen zu können.

13. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die Probenladevorrichtung ferner ein Vakuumgefäß (6) einschließt, in dem die Probenhaltevorrichtung (2) und die längliche(n) Sonde oder Sonden (3) verschiebbar montiert ist/sind, wobei das Vakuumgefäß an die Öffnung (52) der Vakuumkammerwand anschließbar ist.

14. Verfahren zum Laden einer Probe (1) in den Arbeitsbereich (20) der kryogenfreien Kühlvorrichtung nach Ansprüchen 5 und 13, wobei das Verfahren umfasst:

Platzieren einer Probe (1) in oder auf das Probenhaltegerät (2);
Befestigen des Vakuumgefäßes (6) der Probenladevorrichtung an die Vakuumkammer (4) und ausgerichtet mit der Öffnung (52) der Vakuumkammer;
Evakuieren des Vakuumgefäßes (6);
Öffnen der Öffnung (52) der Vakuumkammer (4) und Betätigen der oder jeder länglichen Sonde (3), um das Probenhaltegerät (2) durch die geöffnete Öffnung einzuführen, sodass das Probenhaltegerät thermisch an den kalten Körper gekoppelt ist;
Ermöglichen, dass die Probe (1) im oder auf dem Probenhaltegerät (2) infolge von Wärmeleitung zum kalten Körper gekühlt wird;

Trennen des Probenhaltegeräts (2) vom kalten Körper; und
Betätigen der oder jeder länglichen Sonde (3), um das Probenhaltegerät (2) in den Arbeitsbereich (20) einzuführen.

15. Verfahren nach Anspruch 14, wobei die Vorrichtung ferner ein zweites Wärmestrahlungsschild (10) umfasst, das innerhalb des ersten Wärmestrahlungsschilds (54) positioniert ist und den Arbeitsbereich (20) umgibt, wobei das kryogenfreie Kühlsystem eine zweite Kühlstufe, kälter als die erste Kühlstufe, aufweist, die an das zweite Wärmestrahlungsschild gekoppelt ist, wobei das zweite Wärmestrahlungsschild eine Öffnung (58) aufweist, die mit den Öffnungen des ersten Wärmestrahlungsschilds und der Vakuumkammerwand (52, 54) ausgerichtet ist, um dem Probenhaltegerät (2) zu erlauben dort hindurch zu gehen, wodurch das Probenhaltegerät für Wärmeleitung lösbar an das zweite Wärmestrahlungsschild gekoppelt werden kann, und wobei vor dem Erreichen des Arbeitsbereichs, das Probenhaltegerät thermisch an das zweite Wärmestrahlungsschild gekoppelt wird, gekühlt durch Erlauben, dass Wärme zum zweiten Strahlungsschild, getrennt vom zweiten Strahlungsschild, fließt und das Probenhaltegerät danach in den Arbeitsbereich eingeführt wird.

Revendications

1. Appareil de refroidissement sans cryogène comprenant :

une chambre sous vide (4) ;
un premier bouclier anti-rayonnement thermique (54) entourant une zone de travail (20) et situé dans la chambre sous vide (4) ;
un système de refroidissement sans cryogène, par exemple un refroidisseur mécanique, comme un refroidisseur GM, un refroidisseur Stirling, ou un tube pulsé, possédant un étage de refroidissement couplé au premier bouclier anti-rayonnement thermique (54) ;
un corps froid (12) formé par une surface liée à une plaque froide couplée à un étage de refroidissement du système de refroidissement ;
un corps de montage froid (15) situé dans la zone de travail (20) et maintenu à une température inférieure à celle du corps froid (12) ;
des orifices alignés (52, 56) dans le premier bouclier anti-rayonnement thermique (54) et la paroi de la chambre sous vide ;
un appareil de chargement de l'échantillon possédant une ou plusieurs sondes allongées (3) et un dispositif porte-échantillon (2) fixé sur l'une ou plusieurs sondes allongées, l'une ou plusieurs sondes allongées pour l'insertion du dispositif porte-échantillon vers la zone de travail (20) à travers les orifices alignées (52, 56) ; et
un connecteur thermique (18), le dispositif porte-échantillon (2) étant configuré pour être connecté au corps de montage froid (15) par le biais du connecteur, et la sonde allongée, ou chacune des sondes allongées (3), étant couplée de façon amovible au dispositif porte-échantillon pour relâcher le dispositif porte-échantillon lorsque le dispositif porte-échantillon est raccordé au corps de montage froid ;
caractérisé en ce que le dispositif porte-échantillon (2) est couplé de façon amovible, pour la conduction thermique par le biais dudit connecteur (18) au corps froid (12) de façon à pré-refroidir un échantillon (1) sur ou dans le dispositif porte-échantillon avant le raccordement du dispositif porte-échantillon au corps de montage froid (15).

2. Appareil selon la revendication 1, le corps froid étant formé par le premier bouclier anti-rayonnement thermique (54), le connecteur (18) pouvant être couplé de façon amovible pour la conduction thermique au premier bouclier anti-rayonnement thermique.

3. Appareil selon la revendication 2, comprenant en outre un deuxième bouclier anti-rayonnement thermique (10) situé à l'intérieur du premier bouclier anti-rayonnement thermique (54), et entourant la zone de travail (20), le système de refroidissement sans cryogène possédant un deuxième étage de refroidissement, plus froid que le premier étage de refroidissement, couplé au deuxième bouclier anti-rayonnement thermique, le deuxième bouclier anti-rayonnement thermique possédant un orifice (58) aligné avec les orifices (52, 56) du premier bouclier anti-rayonnement thermique et de la paroi de la chambre sous vide de façon à permettre le passage à travers du dispositif porte-échantillon (2), le dispositif porte-échantillon pouvant être couplé de façon amovible pour la conduction thermique au deuxième bouclier anti-rayonnement thermique.

4. Appareil selon la revendication 3, le premier bouclier anti-rayonnement thermique (54) étant maintenu à une température comprise entre 45K et 90K, et le deuxième bouclier anti-rayonnement thermique (10) étant maintenu de préférence à une température inférieure à 6K.

5. Appareil selon une quelconque des revendications précédentes, l'orifice aligné (52) dans la paroi de la chambre sous vide comprenant un système de fermeture (5) comme une soupape à vide.

6. Appareil selon une quelconque des revendications précédentes, le ou chaque orifice aligné (56, 58) dans le premier bouclier anti-rayonnement thermique (54) et le deuxième bouclier anti-rayonnement thermique (10) pouvant être fermée par un système de fermeture respectif (25), comme un ou plusieurs volets flexibles ou volets articulés ou à ressort.

7. Appareil selon une quelconque des revendications précédentes, la ou plusieurs sondes allongées (3) comprenant deux ou plusieurs sondes allongées, chacune étant couplée au dispositif de maintien de l'échantillon.

8. Appareil selon la revendication 7, chaque sonde pouvant tourner sur son axe relativement au dispositif porte-échantillon (2), et la ou chaque sonde étant de préférence filetée (18) à un bout afin de définir le connecteur thermique, le connecteur coopérant avec un filet (19) sur le corps froid afin de réaliser une connexion thermique entre eux.

9. Appareil selon une quelconque des revendications 1 à 7, le connecteur thermique comprenant un ou plusieurs ressorts thermo-conducteurs, comprenant généralement un matériau composite à thermo-conductivité élevée et force de ressort élevée, monté afin de réaliser un contact thermique entre le corps froid et le dispositif porte-échantillon (2).

10. Appareil selon la revendication 3, l'appareil de chargement de l'échantillon pouvant tourner relativement à la chambre sous vide (4) et aux premier et deuxième boucliers anti-rayonnement thermique (10, 54) de façon à s'aligner de manière sélective avec le ou chaque connecteur thermique (18) ou avec l'orifice respectif (52, 56, 58) afin de permettre le passage du dispositif porte-échantillon (2) à travers.

11. Appareil selon une quelconque des revendications précédentes, le dispositif porte-échantillon (2) comprenant un ou plusieurs connecteurs électriques pour permettre la réalisation de connexions électriques et thermiques à un corps froid (15) dans la zone de travail (20).

12. Appareil selon une quelconque des revendications précédentes, le dispositif porte-échantillon (2) comprenant un ou plusieurs connecteurs optiques, comme des connecteurs de fibres optiques pour permettre la réalisation de connexions électriques et thermiques à un corps froid (15) dans la zone de travail (20).

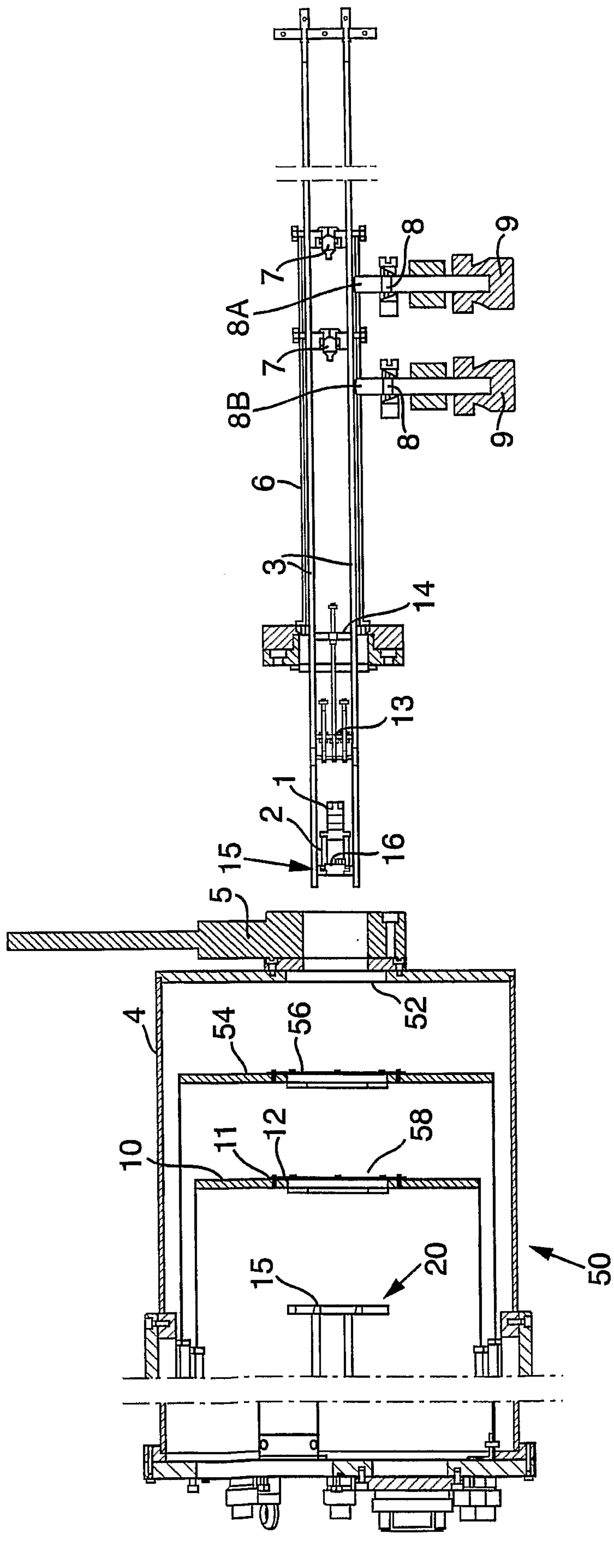
13. Appareil selon une quelconque des revendications précédentes, l'appareil de chargement de l'échantillon comprenant un récipient sous vide (6) dans lequel le dispositif porte-échantillon (2) et la ou les sondes allongées (3) sont montés de façon amovible, le récipient sous vide pouvant être connecté à l'orifice (52) de la paroi de la chambre sous vide.

14. Méthode de maintien d'un échantillon (1) dans la zone de travail (20) d'un appareil de refroidissement sans cryogène selon les revendications 5 et 13, comprenant ce qui suit :

placer un échantillon (1) dans ou sur le dispositif porte-échantillon (2) ;
fixer le récipient sous vide (6) de l'appareil de chargement de l'échantillon sur la chambre sous vide (4) et aligné avec l'orifice (52) de la chambre sous vide ;
évacuer le récipient sous vide (6) ;
ouvrir l'orifice (52) de la chambre sous vide (4) et actionner la ou chaque sonde allongée (3) pour insérer le dispositif porte-échantillon (2) à travers l'orifice ouvert de sorte que le dispositif porte-échantillon soit couplé thermiquement au corps froid ;
permettre le refroidissement de l'échantillon (1) dans ou sur le dispositif porte-échantillon (2) sous l'effet de la conduction thermique au corps froid ;
déconnecter le dispositif porte-échantillon (2) du corps froid ; et
actionner la ou les sondes allongées (3) pour insérer dispositif porte-échantillon (2) dans la zone de travail (20).

15. Méthode selon la revendication 14, l'appareil comprenant en outre un deuxième bouclier anti-rayonnement thermique (10) situé à l'intérieur du premier bouclier anti-rayonnement thermique (54), et entourant la zone de travail (20), le système de refroidissement sans cryogène possédant un deuxième étage de refroidissement, plus froid que le premier étage de refroidissement, couplé au deuxième bouclier anti-rayonnement thermique, le deuxième bouclier anti-rayonnement thermique possédant un orifice (58) aligné avec les orifices du premier bouclier anti-rayonnement thermique et de la paroi de la chambre sous vide (52, 54) de façon à permettre le passage à travers du dispositif porte-échantillon (2), le dispositif porte-échantillon pouvant être couplé de façon amovible pour la conduction thermique au deuxième bouclier anti-rayonnement thermique, et dans lequel, avant d'atteindre la zone de travail, le dispositif porte-échantillon étant couplé thermiquement au deuxième bouclier anti-rayonnement thermique, refroidi en permettant l'écoulement de la chaleur jusqu'au deuxième bouclier anti-rayonnement, déconnecté du deuxième bouclier anti-rayonnement, et le dispositif porte-échantillon étant ensuite inséré dans la zone de travail.

Fig.1.



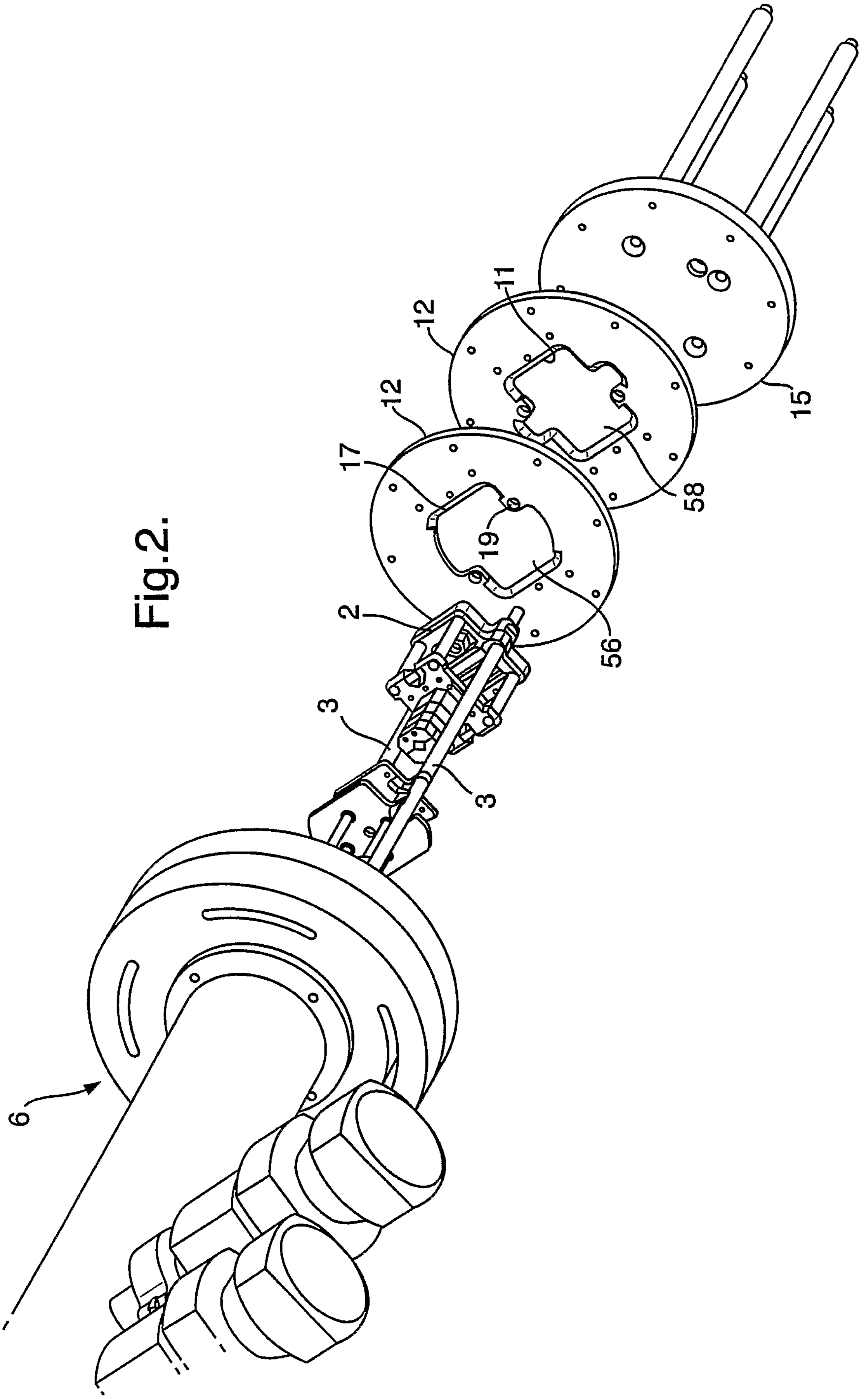


Fig.2.

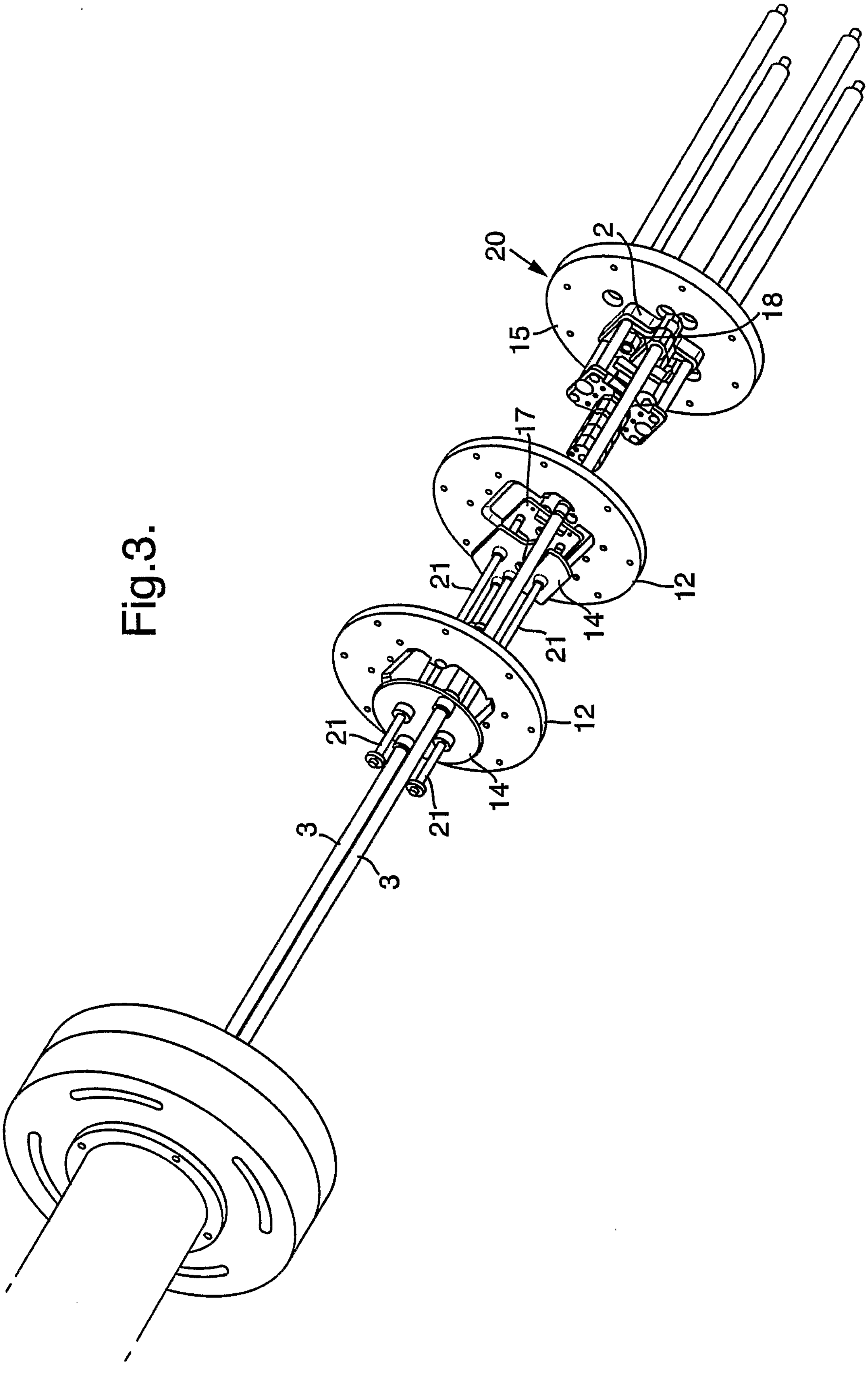


Fig.3.

Fig.4.

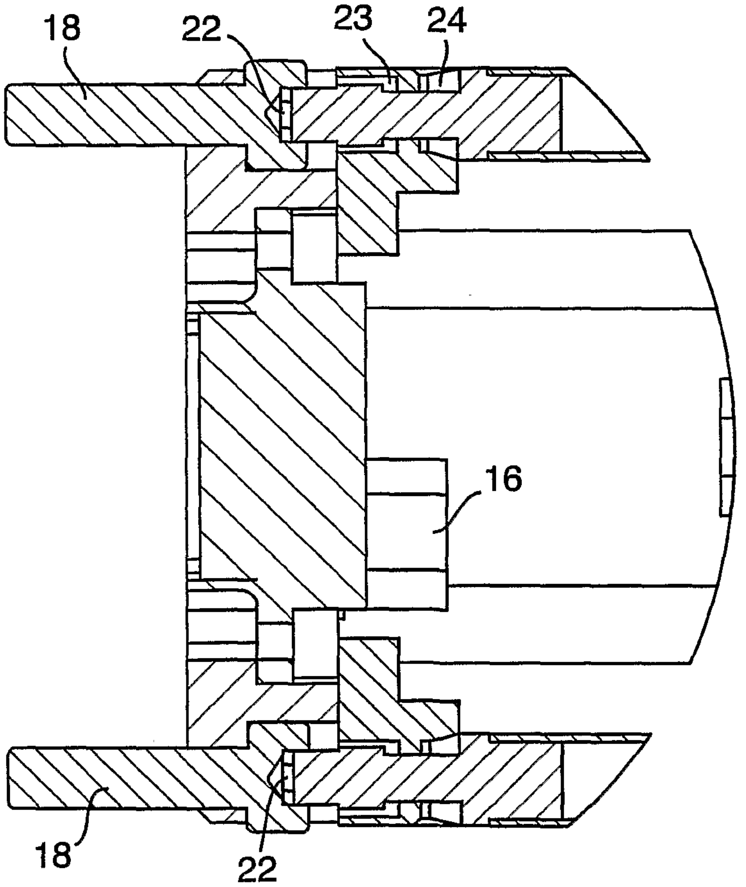


Fig.5.

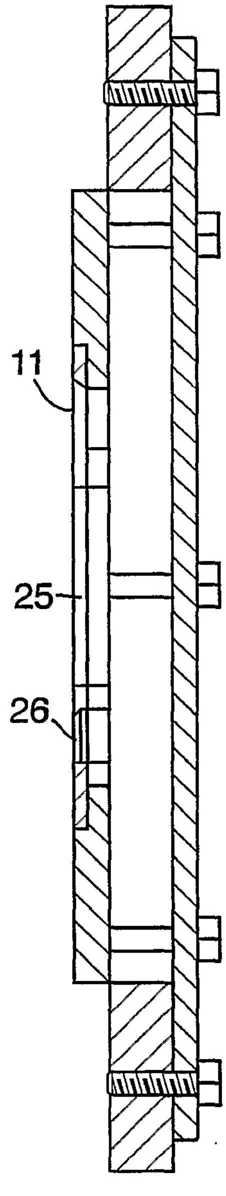


Fig.6.

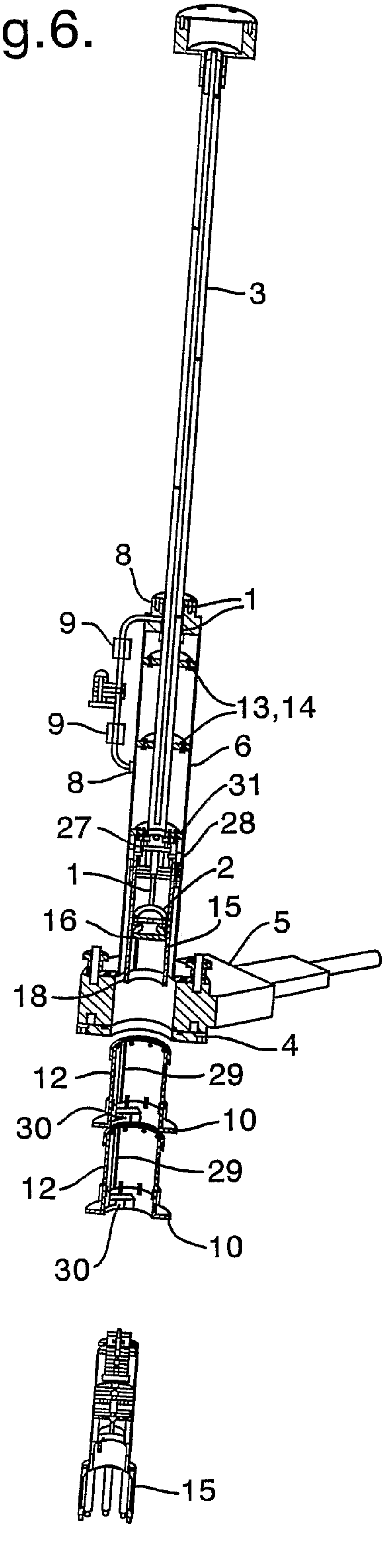
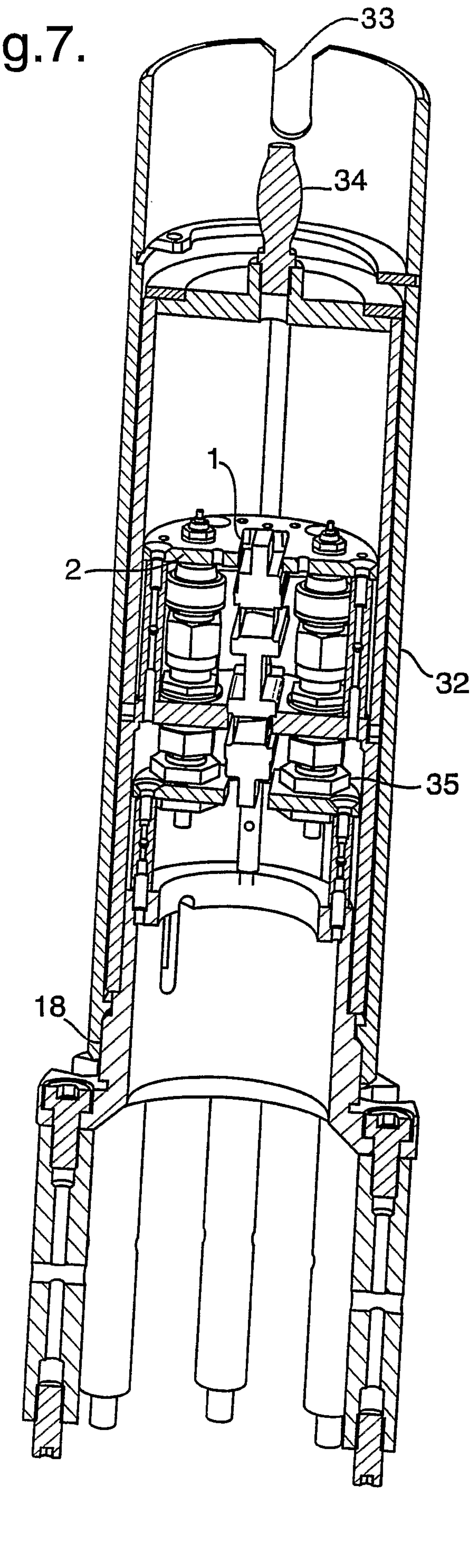


Fig.7.



REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- US 4446702 A **[0004]**
- US 4577465 A **[0004]**
- US 5077523 A **[0004]**
- US 5727392 A **[0004]**
- US 5806319 A **[0004]**
- US 5834938 A **[0004]**
- US 20070234751 A **[0004]**
- US 20080282710 A **[0004]**
- JP 2008014878 A **[0006]**