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(54) **CRYOGEN FREE COOLING APPARATUS AND METHOD**

VORRICHTUNG UND VERFAHREN ZUR KRYOGENFREIEN KÜHLUNG

APPAREIL ET PROCÉDÉ DE REFROIDISSEMENT EXEMPTS DE CRYOGÈNE

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(73) Proprietor: **Oxford Instruments Nanotechnology
Tools Limited
Abingdon, Oxon OX13 5QX (GB)**

(72) Inventors:
• **Garside, John
Abingdon, Oxon OX13 5QX (GB)**
• **Kingley, Simon
Abingdon, Oxon OX13 5QX (GB)**
• **Crowther, Gavin
Abingdon, Oxon OX13 5QX (GB)**
• **Buehler, Matthias
Abingdon, Oxon OX13 5QX (GB)**
• **Wernicke, Doreen
Abingdon, Oxon OX13 5QX (GB)**

(74) Representative: **Gill Jennings & Every LLP
The Broadgate Tower
20 Primrose Street
London EC2A 2ES (GB)**

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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] US-A-5611207 discloses a cryogenic cooling apparatus comprising a vacuum chamber enclosing: a mechanical cooler having first and second heat stations, the second heat station being colder than the first heat station, a cylindrical radiation shield thermally secured to the first heat station and provided with circular access opening, and a heat switch thermally secured to the second heat station. A shutter is rotated to thermally couple a measurement insert to the radiation shield and pre-cool the insert. After pre-cooling the insert, the shutter is again rotated and the insert is thermally coupled to the second heat station. US-A-5611207 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] A cryogen free cooling apparatus and a method of loading a sample into the working region of a cryogen free cooling apparatus according to the present invention is defined in claim 1 and claim 13, respectively.

[0009] 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.

[0010] We have devised a new type of apparatus in which the problems set out above are overcome by utilizing the heat radiation shields within the vacuum chamber to pre-cool a sample before the sample reaches the working region.

[0011] If three or more shields are provided, one or more could be used for precooling.

[0012] 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.

[0013] 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.

[0014] 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.

[0015] 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.

[0016] 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.

[0017] 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 the 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.

[0018] 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.

[0019] 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 according to the current invention;

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

[0020] 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.

[0021] 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.

[0022] 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".

[0023] 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.

[0024] 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 ar-

rangement 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.

[0025] 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.

[0026] 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).

[0027] 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.

[0028] 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.

[0029] 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.

[0030] 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.

[0031] 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.

[0032] 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:

[0033] 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.

[0034] In all embodiments, the connection to the or each cold body can optionally be via thermally conductive spring contacts rather than screw connection.

[0035] In all embodiments, the connection to the radiation shields can optionally be via thermally conductive spring contacts or screw contacts.

[0036] 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.

[0037] 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 aluminum 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.

[0038] 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.

[0039] 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.

[0040] 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.

[0041] 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;
 a cryofree cooling system having a first cooling stage coupled to the first heat radiation shield (54) and a second cooling stage, colder than the first cooling stage;
 aligned apertures (52, 56) in the first heat radiation shield (54) and the 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 (3), the one or more elongate probes for inserting the sample holding device through the aligned apertures (52, 56) to the working region (20); and
 one or more thermal connectors (18), whereby the sample holding device is releasably coupled for heat conduction via said thermal connector(s) to the first heat radiation shield (54) so as to pre-cool a sample (1) on or in the sample holding device before the sample holding device is inserted into the working region (20);
characterised by a second heat radiation shield (10) located inside the first radiation shield (54) and surrounding the working region (20), the second heat radiation shield coupled to the second cooling stage and having an aperture (58) aligned with the apertures (52, 56) of the first heat radiation shield and the vacuum chamber wall so as to allow the sample holding device (2) to pass therethrough, whereby the sample holding device is releasably coupled for heat conduction via said thermal connector(s) (18) to the second heat radiation shield so as to further pre-cool a sample (1) on or in the sample holding device before the sample holding device is inserted into the working region (20); and
 a support (15) located at the working region (20) and held at a lower temperature than the second heat radiation shield, wherein the sample holding device (2) is configured to connect with the support by operation of the probe(s) (3).
2. Apparatus according to claim 1, wherein the aligned aperture (52) in the vacuum chamber wall includes a closure system (5), such as a vacuum valve.
 3. Apparatus according to claims 1 or 2, wherein the aligned apertures (56, 58) in the first and second heat radiation shields (54, 10) include respective closure systems (25).
 4. Apparatus according to claim 3, wherein the respective closure systems for the first and second heat radiation shields (54, 10) comprise one or more flexible flaps or hinged and sprung flaps.
 5. Apparatus according to any of the preceding claims, wherein the sample loading apparatus comprises two or more elongate probes (3), each coupled to the sample holding device (2).
 6. Apparatus according to claim 5, wherein the or each probe (3) is rotatable about its axis relative to the sample holding device (2).
 7. Apparatus according to claim 5 or claim 6, wherein the or each probe (3) is screw threaded at one end to define a said thermal connector (18), the connector cooperating with a screw thread on the first heat radiation shield (54) to achieve a thermal connection therebetween.
 8. Apparatus according to any of claims 1 to 5, wherein one or more of the thermal connector(s) comprises one or more thermally conductive springs (31) fitted to make thermal contact between the first heat radiation shield (54) and the sample holding device (2).
 9. Apparatus according to claim 8, wherein the conductive springs (31) comprise composite material with high thermal conductivity and high spring force.
 10. Apparatus according to any of the preceding claims, wherein the sample loading apparatus is rotatable relative to the vacuum chamber (4) and the heat shields (54, 10) so as selectively to align with the or each thermal connector or with the respective aperture so as to allow the sample holding device (2) to be passed therethrough.
 11. Apparatus according to any of the preceding claims, wherein the apparatus is configured such that the or each probe (3) is releasably coupled to the sample holding device (2) whereby a first operation of the probe(s) causes the sample holding device to be connected to the support (15) at the working region (20), and a second, subsequent operation enables the probe(s) to be released from the sample holding device and retracted.
 12. 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.
 13. A method of loading a sample (1) into the working region (20) of a cryogen free cooling apparatus according to claim 12, 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 first heat radiation shield (54);
 allowing the sample (1) in or on the sample holding device to be cooled as a result of heat conduction to the first heat radiation shield (54);
 disconnecting the sample holding device (2) from the first heat radiation shield (54); and
 operating the or each elongate probe (3) to insert the sample holding device into the working region (20).
14. A method according to claim 13, wherein prior to reaching the working region (20), the sample holding device (2) is thermally coupled to the second heat radiation shield (10), 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 (20).

Patentansprüche

1. Kryogenfreie Kühlvorrichtung, umfassend: neu

Eine Vakuumkammer (4);
 ein erstes Wärmestrahlungsschild (54), das einen Arbeitsbereich (20) umgibt und in der Vakuumkammern positioniert ist;
 ein kryofreies Kühlsystem mit einer ersten Kühlstufe, die an das erste Wärmestrahlungsschild (54) gekoppelt ist und einer zweiten Kühlstufe, die kälter als die erste Kühlstufe ist;
 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 (3) angebracht ist, die eine oder mehrere längliche Sonden zum Einführen des Haltegeräts durch die ausgerichteten Öffnungen (52, 56) in den Arbeitsbereich (20); und
 einen oder mehrere thermischen Anschluss/Anschlüsse (18), wodurch das Probenhaltegerät zur Wärmeleitung über den/die thermischen Anschluss/Anschlüsse lösbar an das erste Wärmestrahlungsschild (54) gekoppelt ist, um eine Probe (1) auf dem oder im Probenhaltegerät vorzukühlen, bevor das Probenhaltegerät in den Arbeitsbereich (20) eingeführt wird;
gekennzeichnet durch ein zweites Wärme-

strahlungsschild (10), das innerhalb des ersten Wärmestrahlungsschilds (54) positioniert ist und den Arbeitsbereich (20) umgibt, wobei das zweite Wärmestrahlungsschild an die zweite Kühlstufe gekoppelt ist und 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 über den/die Anschluss/Anschlüsse (18) lösbar an das zweite Wärmestrahlungsschild gekoppelt ist, um eine Probe (1) auf dem oder im Probenhaltegerät weiter vorzukühlen, bevor das Probenhaltegerät in den Arbeitsbereich (20) eingeführt wird; und
 einen Träger (15), der sich im Arbeitsbereich (20) befindet und auf einer geringeren Temperatur als das zweite Wärmestrahlungsschild gehalten wird, wobei das Probenhaltegerät (2) so ausgelegt ist, dass es durch Betätigung der Sonde(n) (3) mit dem Träger verbunden werden kann.

2. Vorrichtung nach Anspruch 1, wobei die ausgerichtete Öffnung (52) in der Vakuumkammerwand ein Verschlusssystem (5) wie beispielsweise ein Vakuumventil einschließt.

3. Vorrichtung nach Ansprüchen 1 oder 2, wobei die ausgerichteten Öffnungen (56, 58) in den ersten und zweiten Wärmestrahlungsschilden (54, 10) jeweilige Verschlusssysteme (25) einschließen.

4. Vorrichtung nach Anspruch 3, wobei die jeweiligen Verschlusssysteme für die ersten und zweiten Wärmestrahlungsschilden (54, 10) eine oder mehrere flexible Klappen oder scharnierte und gefederte Klappen umfasst.

5. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die Probenladevorrichtung zwei oder mehrere längliche Sonden (3) umfasst, die jeweils an das Probenhaltegerät (2) gekoppelt sind.

6. Vorrichtung nach Anspruch 5, wobei die oder jede Sonde (3) relativ zum Probenhaltegerät (2) um ihre Achse drehbar ist.

7. Vorrichtung nach Anspruch 5 oder Anspruch 6, wobei die oder jede Sonde (3) an einem Ende mit einem Schraubengewinde versehen ist, um einen besagten thermischen Anschluss (18) zu definieren, wobei der Anschluss mit einem Schraubengewinde am ersten Wärmestrahlungsschild (54) kooperiert, um eine thermische Verbindung dazwischen zu erzielen.

8. Vorrichtung nach einem der Ansprüche 1 bis 5, wobei ein thermischer Anschluss oder mehrere der thermischen Anschlüsse eine oder eine oder mehrere thermisch leitfähige Federn (31) umfasst/umfassen, die zur Herstellung von thermischen Kontakt zwischen dem ersten Wärmestrahlungsschild (54) und dem Probenhaltegerät (2) angebracht sind. 5
9. Vorrichtung nach Anspruch 8, wobei die leitfähigen Federn (31) Verbundmaterial mit hoher thermischer Leitfähigkeit und hoher Federkraft umfassen. 10
10. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die Probenladevorrichtung relativ zur Vakuumkammer (4) und den Wärmeschilden (54, 10) drehbar ist, um sich selektiv mit dem oder jedem thermischen Anschluss oder mit der jeweiligen Öffnung auszurichten, um dem Probenhaltegerät (2) zu erlauben, dort hindurch geleitet zu werden. 15
11. Vorrichtung nach einem vorhergehenden Anspruch, wobei die Vorrichtung derart ausgelegt ist, dass die oder jede Sonde (3) lösbar an das Probenhaltegerät (2) gekoppelt ist, wodurch ein erster Arbeitsgang der Sonde(n) bewirkt, dass das Probenhaltegerät mit dem Träger (15) im Arbeitsbereich (20) verbunden wird und ein zweiter, anschließender Arbeitsgang der/den Sonde(n) ermöglicht, aus dem Probenhaltegerät freigegeben und zurückgezogen zu werden. 25
12. Vorrichtung nach einem der vorhergehenden Ansprüche, wobei die Probenladevorrichtung ferner ein Vakuumgefäß (6) einschließt, in das Probenhaltegerät (2) und die längliche(n) Sonde oder Sonden (3) verschiebbar montiert ist/sind, wobei das Vakuumgefäß an die Öffnung (52) der Vakuumkammerwand anschließbar ist. 30
13. Verfahren zum Laden einer Probe (1) in den Arbeitsbereich (20) einer kryogenfreien Kühlvorrichtung nach Anspruch 12, wobei das Verfahren umfasst 35
- Platzieren einer Probe (1) in oder auf das Probenhaltegerät (2); 40
- Befestigen des Vakuumgefäßes (6) der Probenladevorrichtung an die Vakuumkammer (4) und ausgerichtet mit der Öffnung (52) der Vakuumkammer; 45
- Evakuieren des Vakuumgefäßes (6); 50
- 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 das erste Wärmestrahlungsschild (54) gekoppelt ist; 55
- Ermöglichen, dass die Probe (1) im oder auf dem Probenhaltegerät, infolge von Wärmeleitung zum ersten Wärmestrahlungsschild (54)

gekühlt wird;
Trennen des Probenhaltegeräts (2) vom ersten Wärmestrahlungsschild (54); und
Betätigen der oder jeder länglichen Sonde (3), um das Probenhaltegerät in den Arbeitsbereich (20) einzuführen.

14. Verfahren nach Anspruch 13, wobei das Probenhaltegerät (2), vor Erreichen des Arbeitsbereichs (20), thermisch an das zweite Wärmestrahlungsschild (10) gekoppelt wird, gekühlt wird durch Erlauben, dass Wärme zum zweiten Strahlungsschild fließt, vom zweiten Strahlungsschild getrennt wird, und das Probenhaltegerät danach in den Arbeitsbereich (20) eingeführt wird.

Revendications

1. Un 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 ;
un système de refroidissement sans cryogène comportant un premier étage de refroidissement raccordé au premier bouclier anti-rayonnement thermique (54) et un deuxième étage de refroidissement, plus froid que le premier étage de refroidissement ;
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 la ou les sondes allongées (3), cette ou ces sondes allongées est ou sont conçue(s) pour l'insertion du dispositif porte-échantillon au travers des orifices alignés (52, 56), jusqu'à la zone de travail (20) ; et
un ou plusieurs connecteurs thermiques (18), le dispositif porte-échantillon étant configuré pour une connexion amovible pour assurer une conduction thermique par le biais dudit ou desdits connecteur(s) thermique(s) au niveau du premier bouclier anti-rayonnement thermique (54) de façon à pré-refroidir un échantillon (1) sur ou dans le dispositif porte-échantillon avant l'insertion du dispositif porte-échantillon dans la zone de travail (20) ;
caractérisé par un deuxième bouclier anti-rayonnement thermique (10) implanté à l'intérieur du premier bouclier anti-rayonnement thermique (54) et entourant la zone de travail (20), et ce deuxième bouclier anti-rayonnement ther-

- mique est raccordé au deuxième étage de refroidissement et a un orifice (58) aligné sur 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 au travers du dispositif porte-échantillon (2), et ce dispositif porte-échantillon peut être raccordé, de façon amovible, pour assurer une conduction thermique par le biais dudit ou desdits connecteur(s) thermique(s) (18) au deuxième bouclier anti-rayonnement thermique de façon à assurer un pré-refroidissement supplémentaire d'un échantillon (1) sur ou dans le dispositif porte-échantillon avant que ce dispositif porte-échantillon ne soit inséré dans la zone de travail (20) ; et un support (15) implanté au niveau de la zone de travail (20) et maintenu à une température inférieure à celle du deuxième bouclier anti-rayonnement thermique, de telle sorte que le dispositif porte-échantillon (2) est configuré de manière à soutenir le fonctionnement de la ou des sondes (3).
2. L'appareil que décrit la revendication 1, si ce n'est que l'orifice aligné (52) dans la paroi de la chambre sous vide comporte un système de fermeture (5), comme, par exemple, une soupape à vide.
 3. L'appareil que décrit la revendication 1 ou 2, si ce n'est que les orifices alignés (56, 58) dans les premier et deuxième boucliers anti-rayonnement thermique (54, 10) comportent des systèmes respectifs de fermeture (25).
 4. L'appareil que décrit la revendication 3, si ce n'est que les systèmes respectifs de fermeture pour les premier et deuxième boucliers anti-rayonnement thermique (54, 10) comportent un ou plusieurs volets flexibles ou volets à charnières et à ressort.
 5. L'appareil que décrit l'une ou l'autre des revendications précédentes, si ce n'est que l'appareil de chargement de l'échantillon comporte au moins deux sondes allongées (3), et chacune de ces sondes est raccordée au dispositif porte-échantillon (2).
 6. L'appareil que décrit la revendication 5, si ce n'est que la ou les sondes (3) peuvent tourner autour de leur axe par rapport au dispositif porte-échantillon (2).
 7. L'appareil que décrit la revendication 5 ou 6, si ce n'est que la ou les sondes (3) est ou sont filetée(s) à un bout afin de définir un dit connecteur thermique (18), le connecteur coopérant avec un filet sur le premier bouclier anti-rayonnement thermique (54) afin de réaliser une connexion thermique entre eux.
 8. L'appareil que décrit l'une ou l'autre des revendications 1 à 5, si ce n'est qu'au moins un des connecteurs thermiques comporte un ou plusieurs ressorts thermo-conducteurs (31) implantés de manière à réaliser un contact thermique entre le premier bouclier anti-rayonnement thermique (54) et le dispositif porte-échantillon (2).
 9. L'appareil que décrit la revendication 8, si ce n'est que les ressorts conducteurs (31) se composent d'un matériau composite à forte thermo-conductivité et à force élevée de ressort.
 10. L'appareil que décrit l'une ou l'autre des revendications précédentes, si ce n'est que l'appareil de chargement de l'échantillon peut tourner par rapport à la chambre sous vide (4) et aux boucliers anti-rayonnement thermique (54, 10) de façon à s'aligner de manière sélective sur le ou les connecteurs thermiques ou sur l'orifice respectif afin d'y permettre le passage du dispositif porte-échantillon (2).
 11. L'appareil que décrit l'une ou l'autre des revendications précédentes, si ce n'est que cet appareil est configuré de telle sorte que la ou les sondes (3) est ou sont raccordée(s), de manière amovible, au dispositif porte-échantillon (2) de telle sorte qu'un premier fonctionnement de cette ou ces sondes entraîne le raccordement de ce dispositif porte-échantillon au support (15) au niveau de la zone de travail (20) et qu'un deuxième fonctionnement et des fonctionnements ultérieurs permettent à cette ou ces sondes de s'écarter de ce dispositif porte-échantillon et de prendre la position escamotée.
 12. L'appareil que décrit l'une ou l'autre des revendications précédentes, si ce n'est que l'appareil de chargement de l'échantillon comporte, en outre, un récipient sous vide (6) dans lequel le dispositif porte-échantillon (2) et la ou les sondes allongées (3) sont montés, de façon amovible, et ce récipient sous vide peut se raccorder à l'orifice (52) de la paroi de la chambre sous vide.
 13. Une méthode de chargement d'un échantillon (1) dans la zone de travail (20) de l'appareil de refroidissement sans cryogène que décrit la revendication 12, et cette méthode comporte les éléments suivants :
 - le placement d'un échantillon (1) dans ou sur le dispositif porte-échantillon (2) ;
 - la fixation du récipient sous vide (6) de l'appareil de chargement de l'échantillon sur la chambre sous vide (4) et son alignement sur l'orifice (52) de la chambre sous vide ;
 - l'évacuation du récipient sous vide (6) ;
 - l'ouverture de l'orifice (52) de la chambre sous

vide (4) et l'actionnement de la ou des sondes allongées (3) pour insérer le dispositif porte-échantillon (2) dans l'orifice ouvert de manière à raccorder thermiquement ce dispositif porte-échantillon au premier bouclier anti-rayonnement thermique (54); 5
le refroidissement de l'échantillon (1) dans ou sur le dispositif porte-échantillon sous l'effet de la conduction thermique, jusqu'au bouclier anti-rayonnement thermique (54) ; 10
la déconnexion du dispositif porte-échantillon (2) depuis le premier bouclier anti-rayonnement thermique (54) ; et
l'actionnement de la ou des sondes allongées (3) pour insérer le dispositif porte-échantillon dans la zone de travail (20). 15

14. La méthode que décrit la revendication 13, si ce n'est, avant d'atteindre la zone de travail (20), que le dispositif porte-échantillon (2) est raccordé thermiquement au deuxième bouclier anti-rayonnement thermique (10), est refroidi en permettant à la chaleur de circuler jusqu'au deuxième bouclier anti-rayonnement, est déconnecté du deuxième bouclier anti-rayonnement et le dispositif porte-échantillon est ensuite inséré dans la zone de travail (20). 20 25

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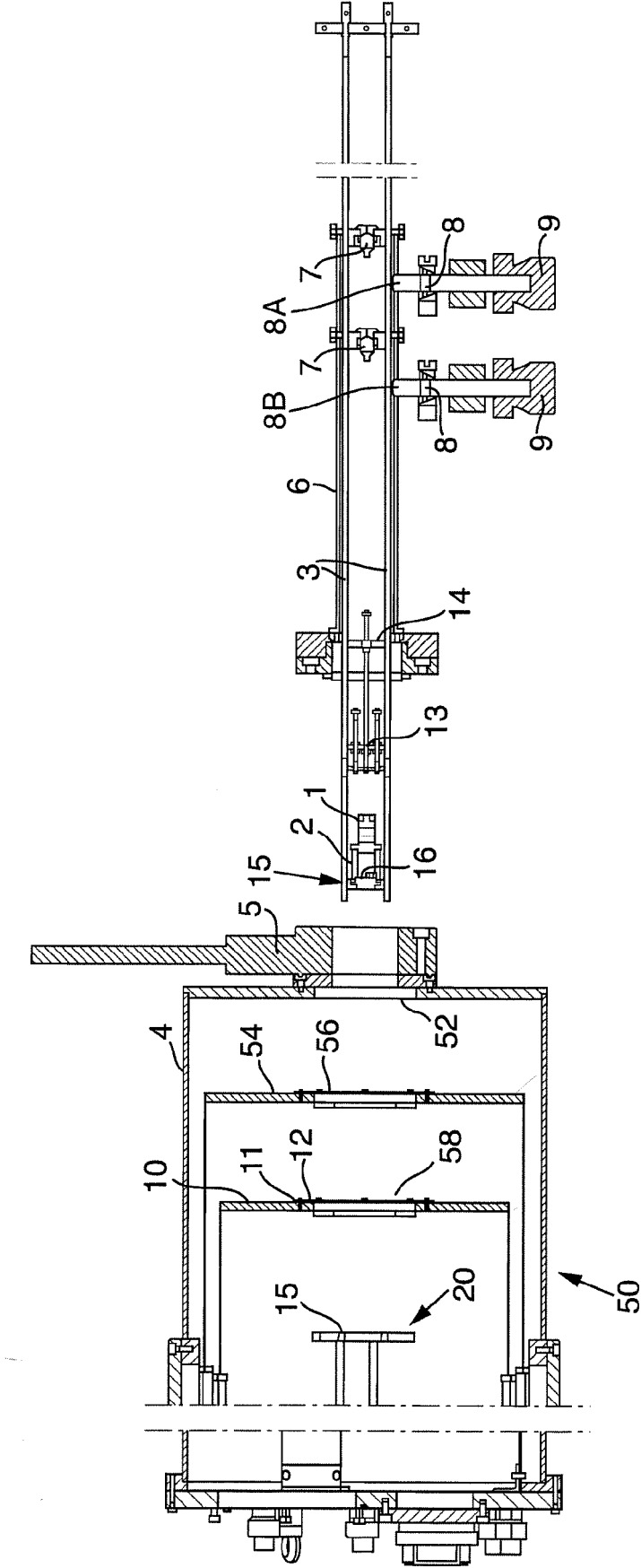
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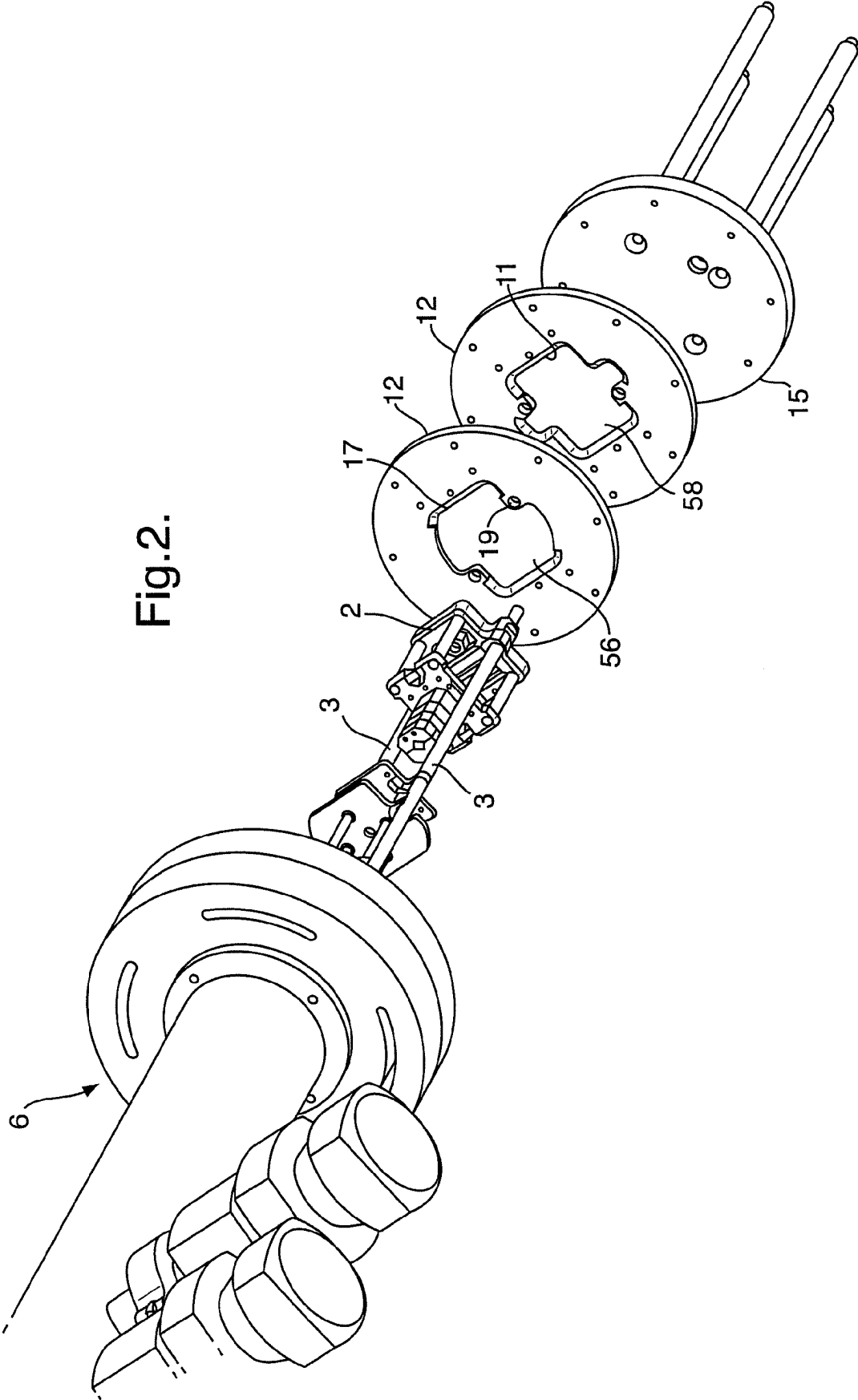
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Fig.1.





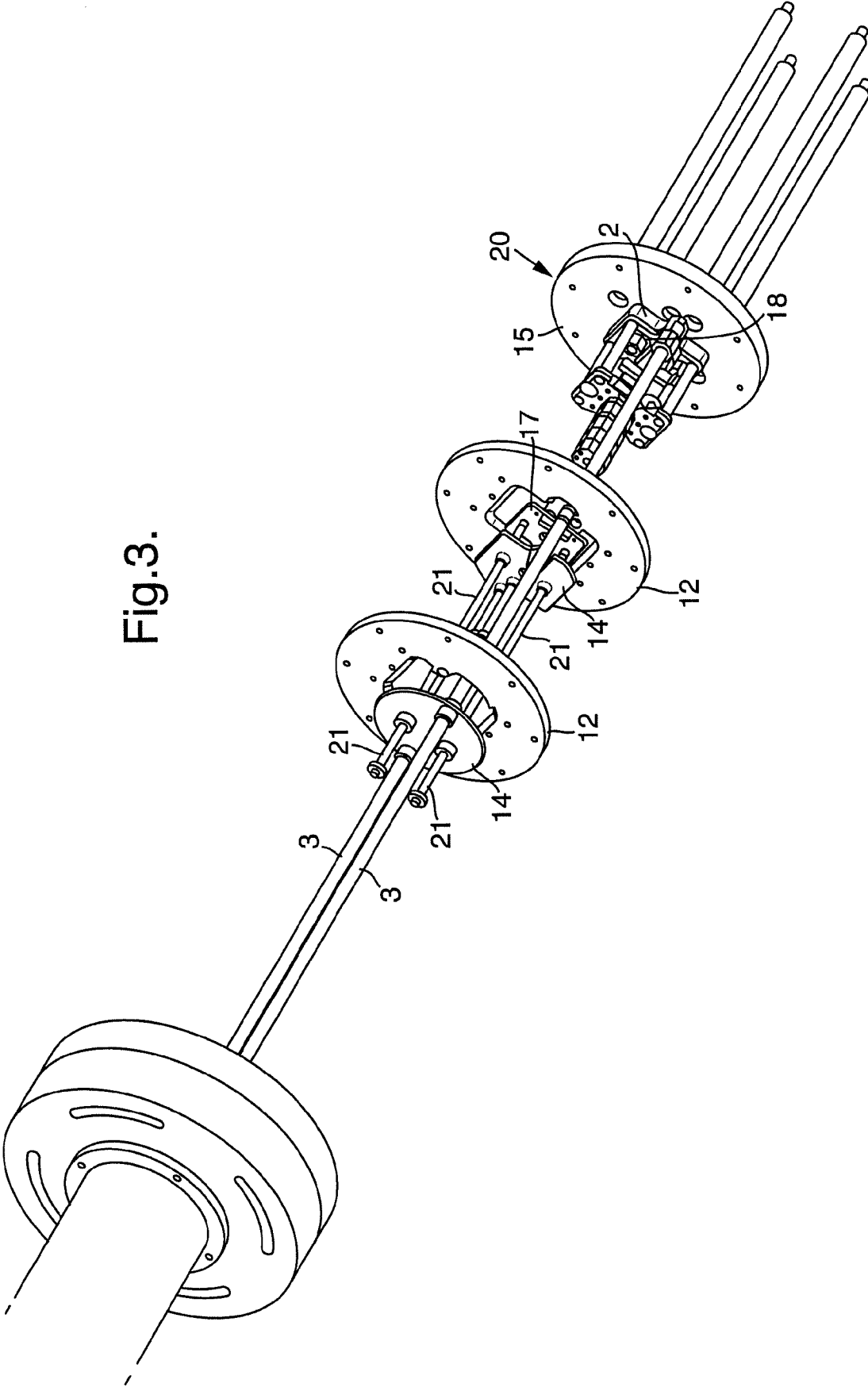


Fig.4.

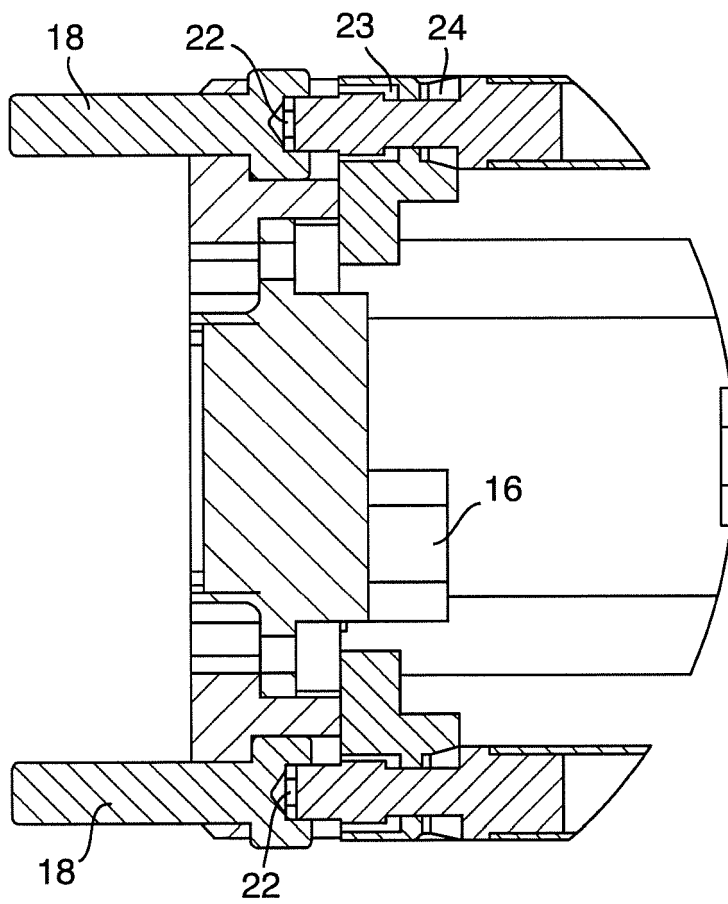


Fig.5.

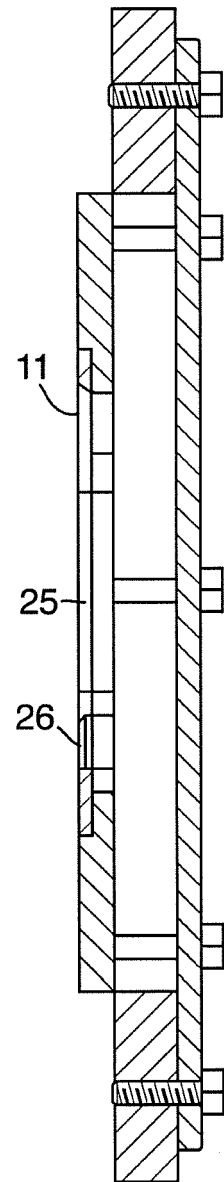


Fig.6.

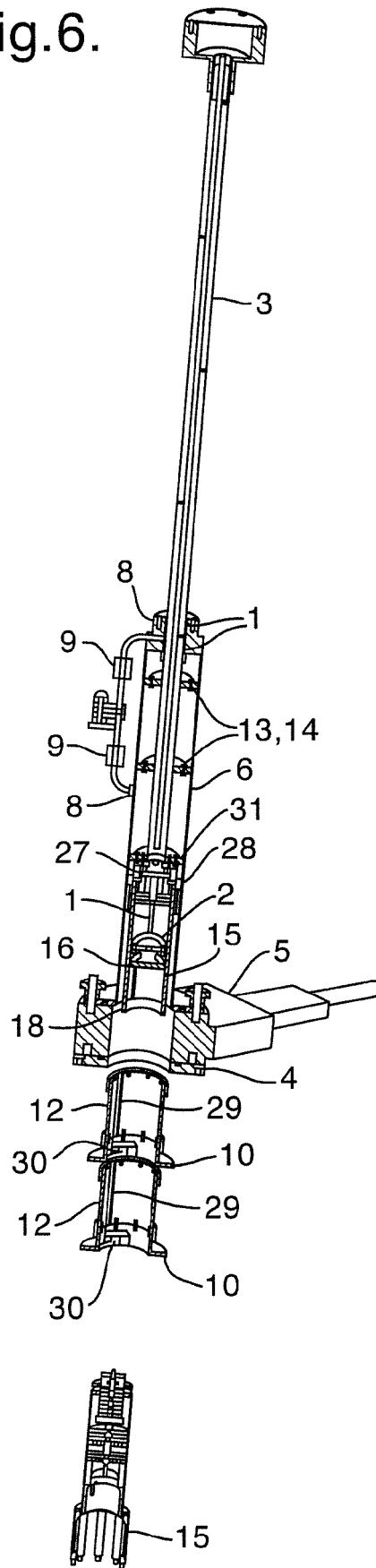
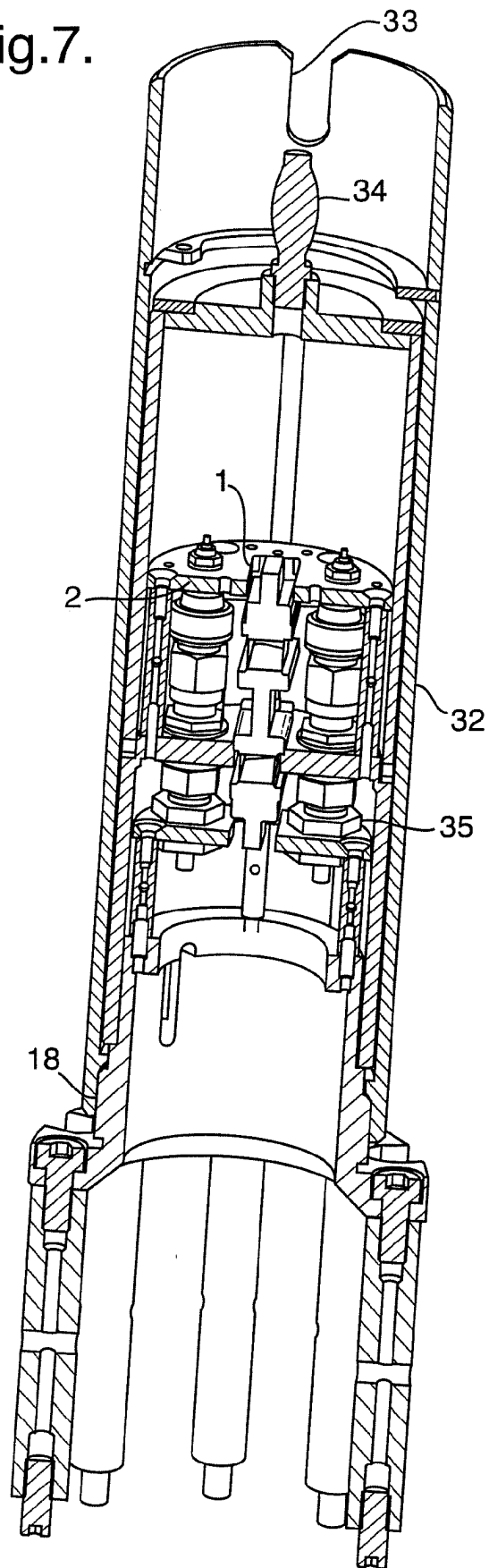


Fig.7.



REFERENCES CITED IN THE DESCRIPTION

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