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

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(57) **ABSTRACT**

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

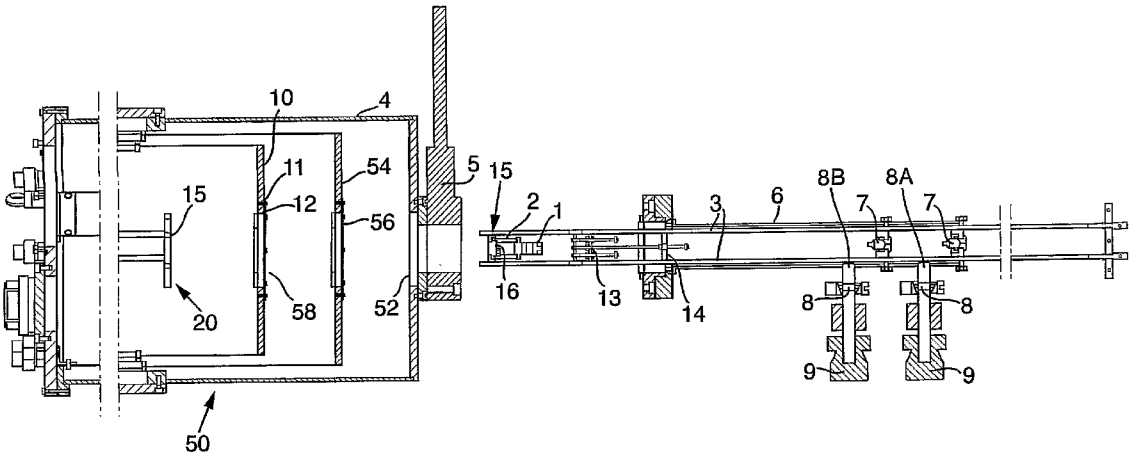
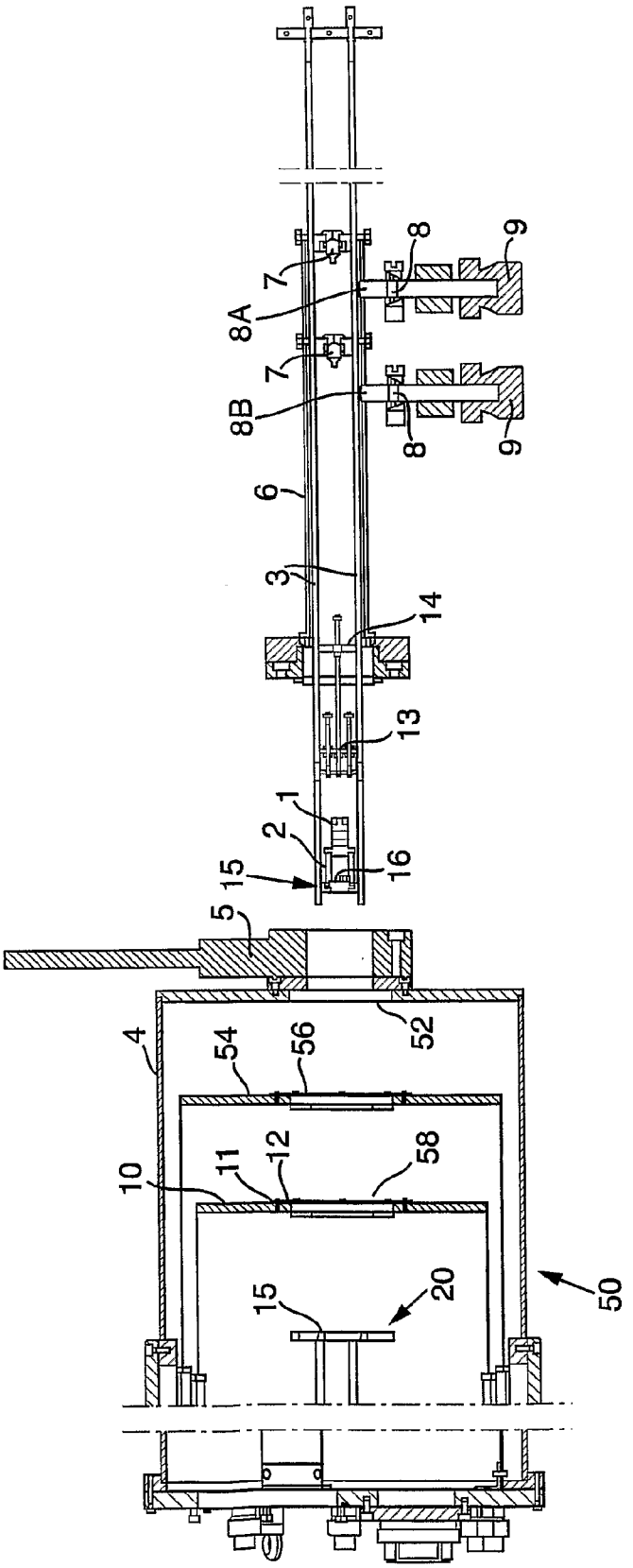


Fig.1.



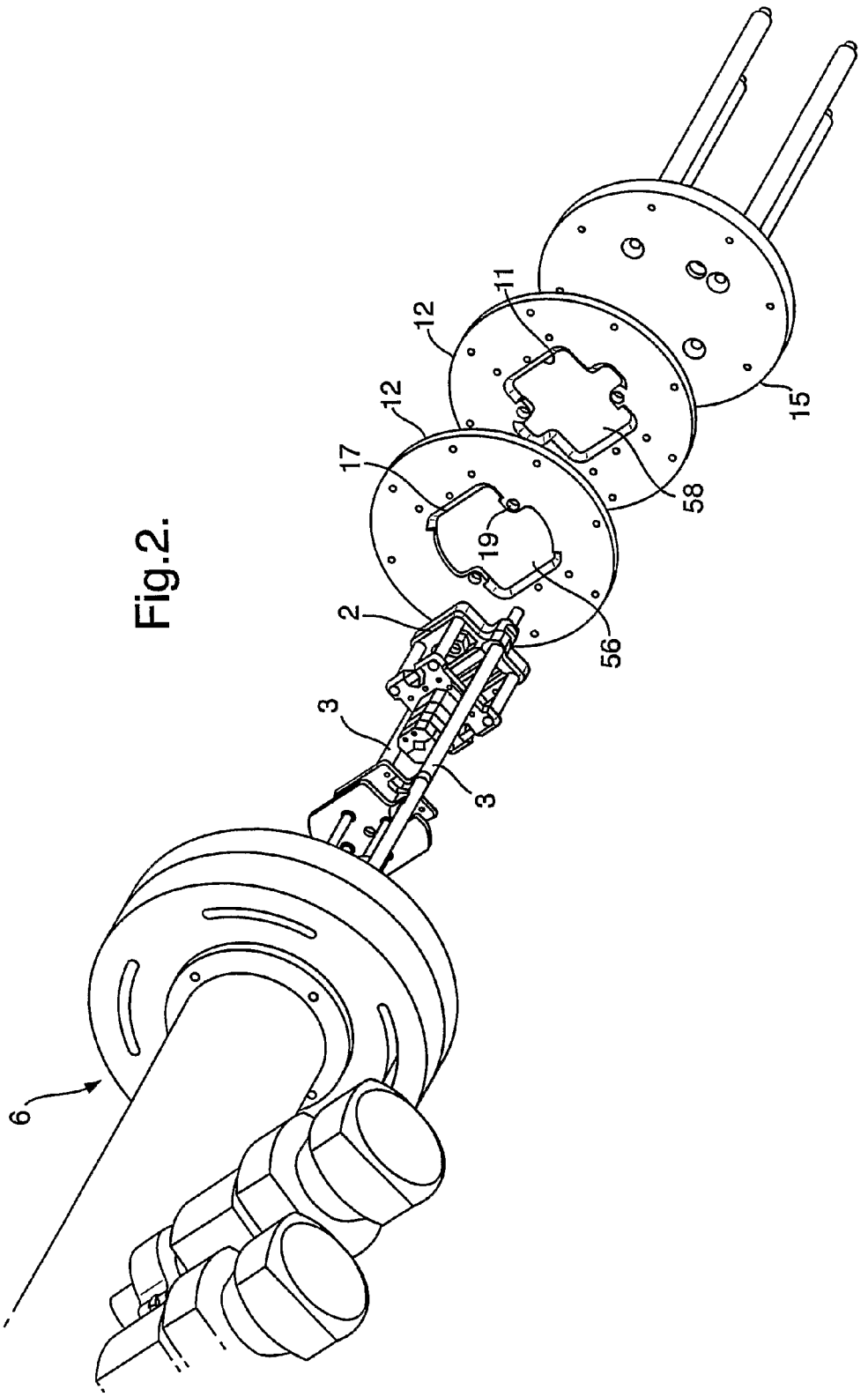


Fig.2.

Fig.3.

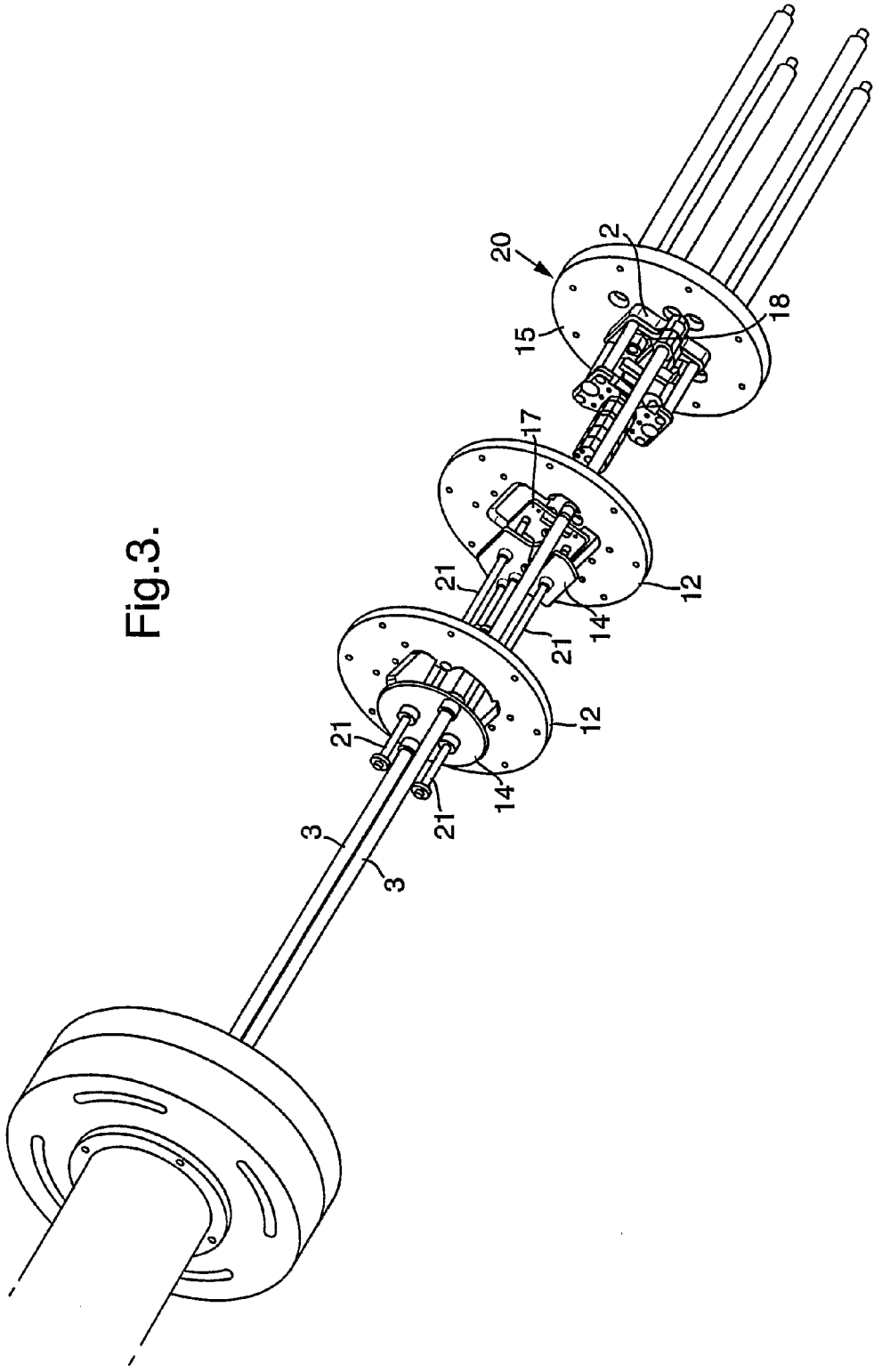


Fig.4.

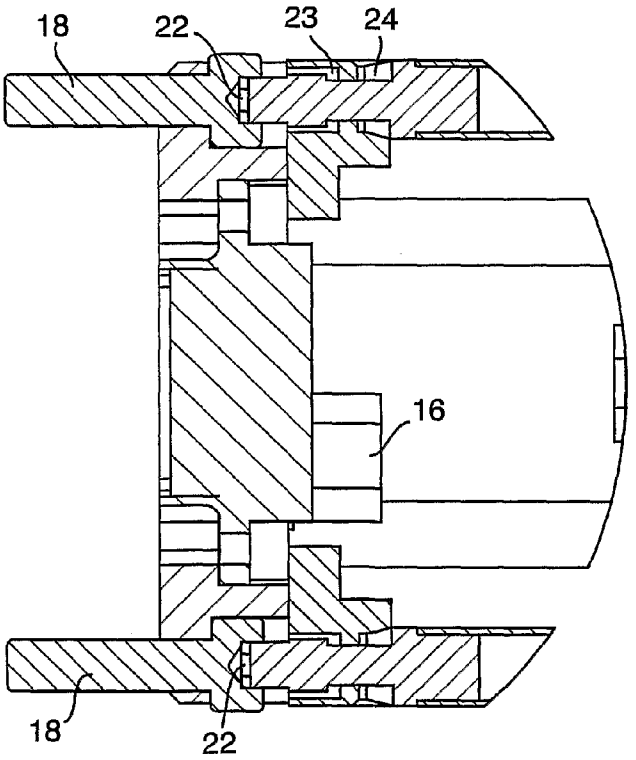


Fig.5.

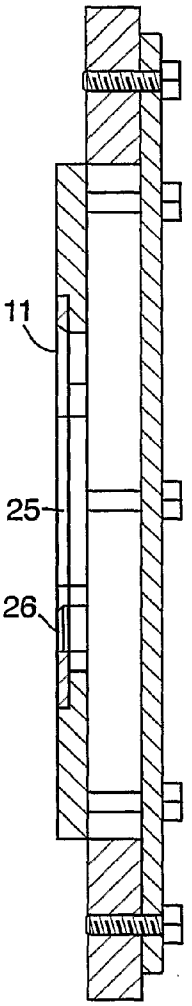


Fig.6.

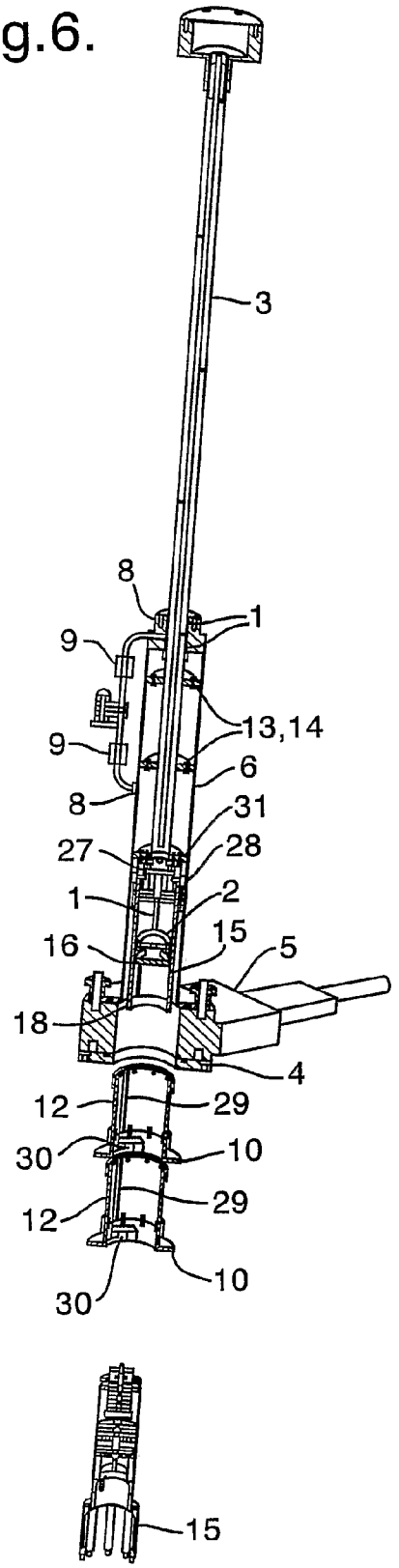
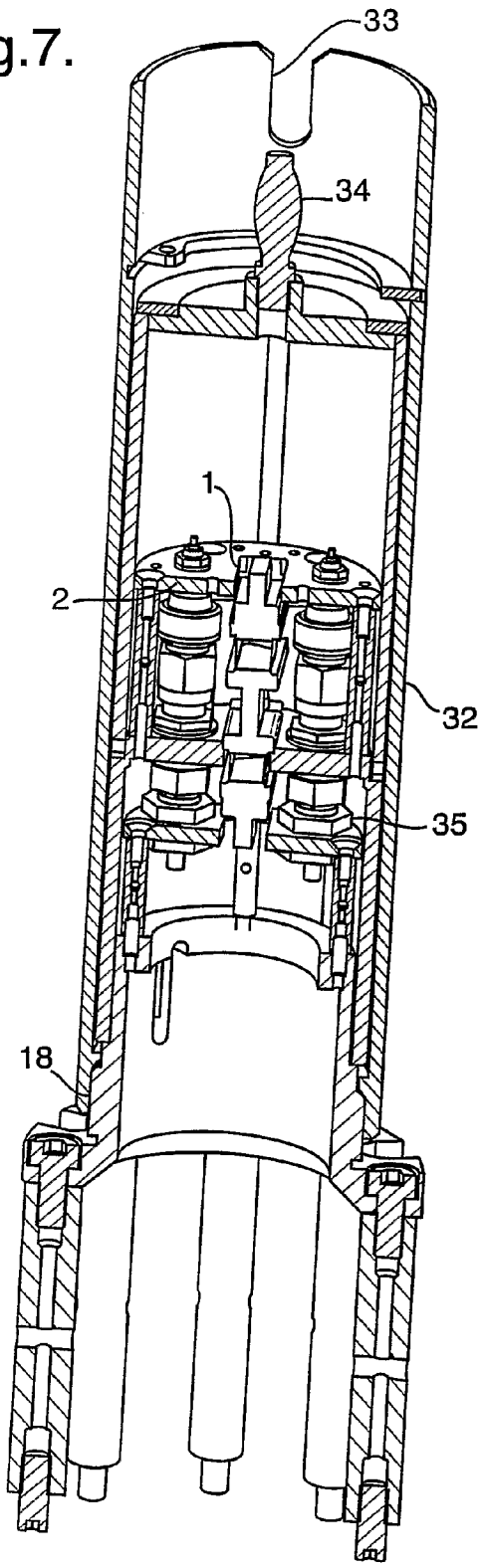


Fig.7.



CRYOGEN FREE COOLING APPARATUS AND METHOD

[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 U.S. Pat. No. 4,446,702, U.S. Pat. No. 4,577,465, U.S. Pat. No. 5,077,523, U.S. Pat. No. 5,727,392, U.S. Pat. No. 5,806,319, U.S. Pat. No. 5,834,938, US-A-20070234751 and US-A-20080282710.

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

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

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

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

[0009] placing a sample in or on the sample holding device;

[0010] securing the vacuum vessel of the sample loading apparatus to the vacuum chamber and aligned with the aperture of the vacuum chamber;

[0011] evacuating the vacuum vessel;

[0012] opening the aperture of the vacuum chamber and operating the or each elongate probe to insert the sample holding device through the opened aperture so that the sample holding device is thermally coupled to a cold body;

[0013] allowing the sample in or on the sample holding device to be cooled as a result of heat conduction to the cold body; and either

[0014] a) disconnecting the sample holding device from the cold body; and operating the or each elongate probe to insert the sample holding device into the working region, or

[0015] b) strengthening the thermal connection to the cold body to increase heat flow away from the sample and further cool the sample.

[0016] 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.

[0017] Although various cold bodies within the cryostat could be used, such as any cold surface coupled to the cooling stage of the cooling system or to an intermediate stage of a sub 4K cooler such as a still of a dilution refrigerator, it is most convenient to utilize the heat radiation shield already present. A further possibility is that the cold body is a body held at the lowest temperature within the cooling apparatus in which case the thermal connector allows a weak thermal connection initially to the cold body for precooling following which the connection to the cold body is strengthened so the sample cools to the desired final temperature.

[0018] 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.

[0019] 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.

[0020] 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.

[0021] 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.

[0022] 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.

[0023] 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.

[0024] 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.

[0025] 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.

[0026] In a particularly preferred embodiment, the or each probe is releasably coupled to the sample holding device whereby a first operation of the probe(s) causes the sample holding device to be connected to a support at the working region, and a second operation enables the probe(s) to be released from the sample holding device and retracted. This enables the probe(s) to be removed from the vacuum chamber of the cryostat so as to reduce heat flow into the cryostat. Actuators to allow this could be provided on the probe or cold body.

[0027] 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.

[0028] It should be understood that the sample loading apparatus just described, particularly the releasable nature of the sample holding device, is considered inventive in its own right and separate from the concept of pre-cooling the sample as defined in the first and second aspects of the invention.

[0029] Some examples of apparatus and methods according to the invention will now be described with reference to the accompanying drawings, in which:

[0030] FIG. **1** shows a cutaway, part sectional view of a first embodiment of the sample loading apparatus;

[0031] FIG. **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);

[0032] FIG. **3** shows a view similar to FIG. **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);

[0033] FIG. **4** shows a detail sectional view of the first embodiment of the sample loading apparatus;

[0034] FIG. **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);

[0035] FIG. **6** shows a cutaway view of a second embodiment of the sample loading apparatus; and

[0036] FIG. **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

[0037] A first embodiment of the current invention is shown in more detail in FIGS. **1** to **5**. In FIG. **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 **8**A 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**.

[0038] The cryostat **50** comprises an outer vacuum vessel **4** which is closed except for a port **52** covered by a large diameter gate valve **5**. Within the vacuum vessel **4** is located a first radiation shield **54** having an aperture **56** aligned with the

aperture **52** of the vacuum chamber, and within the first radiation shield **54** is located a second radiation shield **10** having an aperture **58** aligned with the apertures **52**,**56**. The radiation shields **10**,**54** surround a working region **20** at which is located a cold mounting body **15**.

[0039] 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".

[0040] As can be seen in FIG. **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**.

[0041] Optionally, the apertures **56**,**58** can be closed by a suitable closure mechanism. FIG. **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.

[0042] 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 **8**A,**8**B 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.

[0043] FIG. **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** (FIG. **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).

[0044] 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. FIG. **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.

[0045] FIG. **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.

[0046] 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.

[0047] A second embodiment of the current invention is shown in FIG. **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.

[0048] 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.

[0049] FIG. **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

[0050] 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.

[0051] In all embodiments, the connection to the or each cold body can optionally be via thermally conductive spring contacts rather than screw connection.

[0052] In all embodiments, the connection to the radiation shields can optionally be via thermally conductive spring contacts or screw contacts.

[0053] 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.

[0054] In all embodiments, as an alternative to pre-cooling at progressively lower temperatures at radiation shields, the sample and carrier may optionally be pre-cooled by making a weaker thermal contact to a colder temperature body, such as the coldest body. The relative warming of the cryostat and cooling of the sample and carrier can be controlled by design of the thermal contact.

[0055] 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.

[0056] 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.

[0057] 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.

[0058] 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.

[0059] 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.

1. A cryogen free cooling apparatus comprising at least one heat radiation shield surrounding a working region and located in a vacuum chamber; a cryofree cooling system such as a mechanical cooler such as a GM cooler, Stirling cooler or pulse tube device, having a cooling stage coupled to the heat radiation shield; aligned apertures in the heat radiation shield and vacuum chamber wall; sample loading apparatus having a sample holding device attached to one or more elongate probes for inserting the sample holding device through the aligned apertures to the working region; and a thermal connector, whereby the sample holding device can be releasable coupled for heat conduction via said connector to a cold body or cold bodies within the vacuum chamber so as to pre-cool a sample on or in the sample holding device, characterized in that the or each probe is releasably coupled to the sample holding device whereby a first operation of the probe(s)

causes the sample holding device to be connected to a support at the working region, and a subsequent operation enables the probe(s) to be released from the sample holding device and retracted.

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

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

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

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

6. Apparatus according to claim **1**, wherein the first heat radiation shield is held at a temperature of between 45K and 90K.

7. Apparatus according to claim **1**, wherein the cold body is the coldest body within the cooling apparatus, the thermal connector enabling a weak thermal connection to be achieved with the sample holding device and subsequently a stronger thermal connection allowing a greater rate of heat flow.

8. Apparatus according to claim **1**, wherein the vacuum chamber aperture includes a closure system such as a vacuum valve.

9. Apparatus according to claim **1**, wherein the or each heat radiation shield aperture is closable by a respective closure system.

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

11. Apparatus according to claim **1**, wherein the sample loading apparatus comprises two or more elongate probes, each coupled to the sample holding device.

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

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

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

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

16. Apparatus according to claim **1**, wherein the sample loading apparatus is rotatable relative to the vacuum chamber and heat shield(s) so as selectively to align with the or each thermal connector or with the respective aperture so as to allow the sample holding device to be passed therethrough.

17. (canceled)

18. Apparatus according to claim **1** wherein the sample loading apparatus further includes a vacuum vessel in which the sample holding device and elongate probe or probes are movably mounted, the vacuum vessel being connectable to the aperture of the vacuum chamber wall.

19. Apparatus according to claim **1**, wherein the cryogen free cooling system comprises a mechanical cooler such as a GM cooler, Stirling cooler or pulse tube device.

20. Apparatus according to claim **1**, wherein the sample holder includes one or more electrical connectors to allow electrical and thermal connections to be made to a cold body in the working region.

21. Apparatus according to claim **1**, wherein the sample holder includes one or more optical connectors such as fiber optic connectors to allow electrical and thermal connections to be made to a cold body in the working region.

22. A method of loading a sample into the working region of cryogen free cooling apparatus according to claim **18**, the method comprising:

placing a sample in or on the sample holding device;

securing the vacuum vessel of the sample loading apparatus to the vacuum chamber and aligned with the aperture of the vacuum chamber;

evacuating the vacuum vessel;

opening the aperture of the vacuum chamber and operating the or each elongate probe to insert the sample holding device through the opened aperture so that the sample holding device is thermally coupled to a cold body;

allowing the sample in or on the sample holding device to be cooled as a result of heat conduction to the cold body; and either

a) disconnecting the sample holding device from the cold body; and operating the or each elongate probe to insert the sample holding device into the working region, or

b) strengthening the thermal connection to the cold body to increase heat flow away from the sample and further cool the sample, and detaching the sample holding device from the or each elongate probe and withdrawing the or each elongate probe and subsequently closing the closure system of the aperture of the vacuum chamber.

23. A method according to claim **22**, further comprising a second heat radiation shield located inside the first radiation shield and surrounding the working region, the cryogen free cooling system having a second cooling stage, colder than the first cooling stage, coupled to the second heat radiation shield the second radiation shield having an aperture aligned with the apertures of the first heat radiation shield and vacuum chamber wall so as to allow the sample holding device to pass therethrough, whereby the sample holding device can be releasably coupled for heat conduction to the second heat radiation shield acting as the or a cold body, 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.

24. (canceled)

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