Chapter 8
Design and Operation of a Large, Low Background, 50 mK Cryostat for the Cryogenic Dark Matter Search

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Abstract Cryostats that operate below 1 K have additional requirements involving the need for extremely small heat leaks and alternative cooling methods. This chapter describes the design and operation of a cryostat operating at 50 mK for the Cryogenic Dark Matter Search experiment. Included are descriptions of the cooling and thermal insulation system, seal design, fabrication and operations. Valuable data is provided on the thermal conductivity of Kevlar and the calculation of joint conductance. The particular issue of using only radiopure materials in the cryostat construction is also covered. A list of lessons learned from the cryostat operation is provided.

8.1 Introduction

To support the Cryogenic Dark Matter Search (CDMS) experiment, a sub Kelvin cryostat and support system was built and operated at Soudan MN. In addition to the challenges of milliKelvin operation, the cryostat was made with low background material, and was located in an RF shielded clean room one-half mile below the surface at Soudan Underground State Park.

8.2 Physics Detectors and Towers

The goal of CDMS was to detect dark matter, which has been detected through large-scale gravitational interactions. CDMS used cryogenic germanium and silicon detectors, which are capable of detecting weakly interactive dark matter (WIMPs).

WIMPs are detected through their interactions with the nuclei in the germanium. When a nucleus is hit, it recoils, causing the whole germanium crystal to vibrate.

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These vibrations, or phonons, propagate to the surface of the crystal where they heat sensors consisting of thin aluminum traps connected by tungsten meanders. The tungsten is kept at its Tc (critical temperature) and so is an ultra-sensitive thermometer, one capable of sensing temperature changes a fraction of a milliKelvin. The extra energy—as little as 5 keV—the phonons bring raises the tungsten's resistance by a minute amount. When the phonons reach the aluminum, they excite quasi-particle states, which propagate to the tungsten and heat it up.

When the temperature of the tungsten rises so does the resistance of the circuit. This causes the bias current to decrease since the voltage across the tungsten is held constant. The resulting pulse is picked up by SQUID (Superconducting QUantum Interference Device) amplifiers.

There are other particles that go through the detectors besides WIMPs. The CDMS detectors were shielded to minimize the number of these other particles. The detectors are capable of discriminating between most of them and the WIMP signal [3, 17, 22].

The detectors ran in the Soudan Mine in Minnesota, a half-mile underground. The deep site is chosen to shield the detectors from cosmic rays.

Six detector crystals were mounted in each tower assembly (see Fig. 8.1). The towers provided structural support for the detectors and thermal heat sinking at the base temperature. The tower features included heat sinks at 200 mK, 1 and 5 K for the structural supports and signal wiring. Wiring and structural elements in the tower were a major portion of the total heat load.

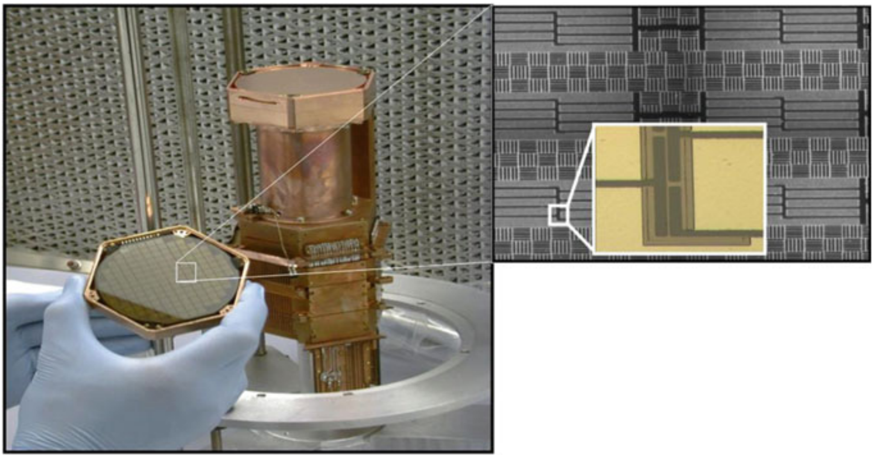

Fig. 8.1 Detector tower with one crystal. Completed tower supports six crystals [17]

8.3 Cryogenic System General Description

Science requirements were for a cryostat with very low radioactive background, shielded against gamma and neutron radiation. Detector operation below the tungsten Tc requires multiple stages of heat shielding. The cryostat had to be large enough to contain seven detector towers. The low background requirement strictly limited materials that could be used.

The CDMS ICEBOX provided the mounting, thermal shielding and cooling for the CDMS detectors. The ICEBOX includes six nested cans with top access, a stem (E-stem) to carry out the signal cables, a stem (C-stem) for heat conduction, and aramid rope suspension. The inner can held 0.022 m^3 volume. The CDMS Icebox at Soudan operated for twelve years including multiple runs over one year in duration.

Figure 8.2 shows the overall layout of the experiment, showing the detector space surrounded by Icebox, a magnetic shield, lead and polyethylene shielding. The assembly was installed in an RF shielded, class 10,000 clean room 2300 ft. below the surface at Soudan Underground State Park [18] in Soudan, MN (Fig. 8.3).

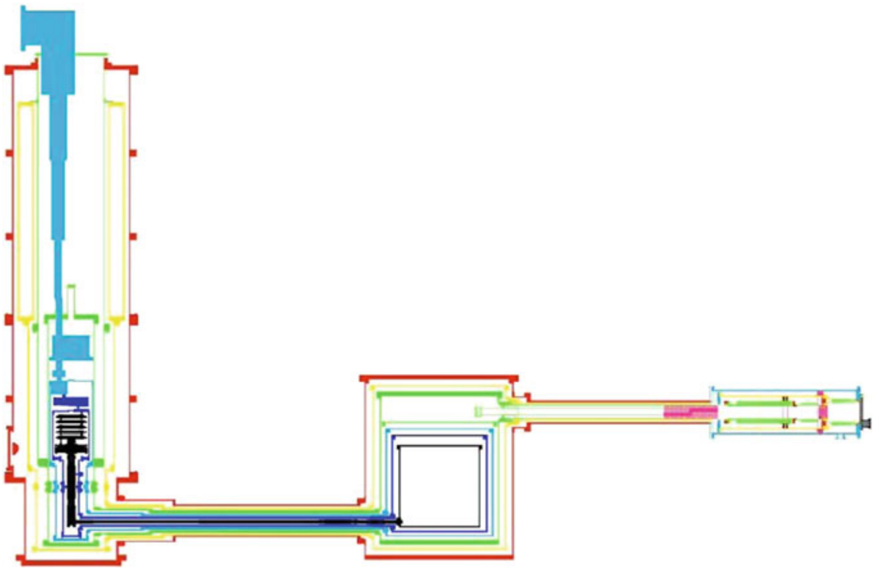

Fig. 8.2 Overall cryogenic layout, dilution refrigerator on the *left*, ICEBOX cans in the *middle*. C-stem provides conductive cooling from the ICEBOX to the fridge. The signal feedthrough is on the *right* [17]

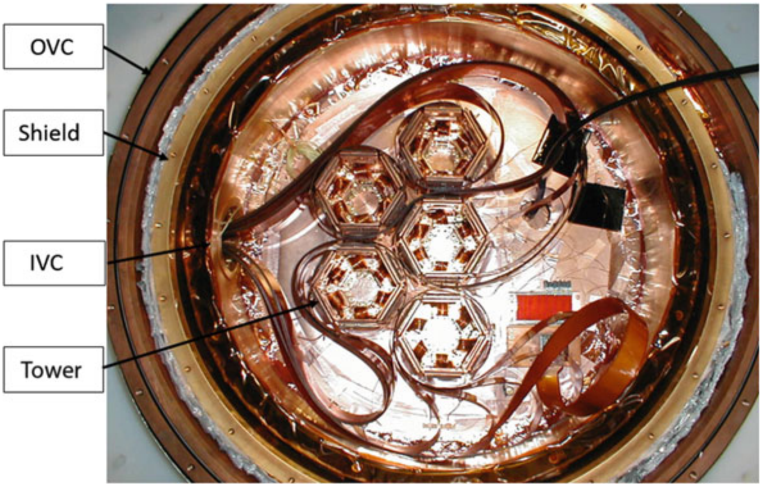

Fig. 8.3 Icebox top, showing tops of the OVC, shield can, IVC can with five towers [17]

8.4 Dilution Refrigerator Introduction

The next sections refer to dilution refrigerator cooling. For the reader unfamiliar with this type of refrigerator the description of a 'wet' can be found in general literature, for example at [7].

8.5 Icebox General Description

The CDMS ICEBOX provided the mounting, thermal shielding and cooling for the CDMS detectors. The ICEBOX includes six nested cans with top access, a stem (E-stem) to carry out the signal cables, a stem (C-stem) for heat conduction, and aramid rope suspension. The stem length was needed to pass through the thicknesses of the lead and polyethylene shielding. Thermal models have confirmed that this number of layers is appropriate for the large milliKelvin detector operation. For CDMS the temperatures chosen were suited to the operating stages of the wet dilution refrigerator. Future large detectors may use dry fridges and may not be limited by helium or nitrogen boiling temperatures.

8.5.1 Icebox Cans

Each of the cans provides heat shielding and mounting for the next inner can. Figure 8.4 shows the top view of the can assembly. The cans are referred to

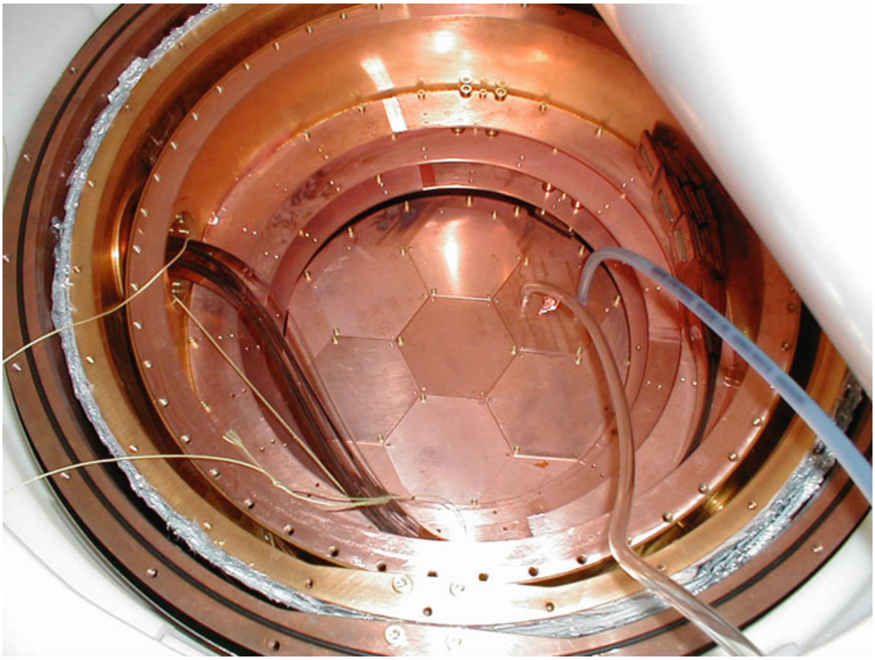

Fig. 8.4 ICEBOX cans, *top view*. The support adjustment screws for the outer layers can be seen at the *bottom* of the view. The signal cables exit through the E-stem on the *left* [3]

(moving from the outermost inward) as the Outer Vacuum Can (OVC), the Shield can (SH), the Inner Vacuum Can (IVC) can, the Still (ST) can, the Cold Plate (CP) can, and the Mixing Chamber (MC) can. The names refer to the associated dilution refrigerator stage.

The MC lid seen in Fig. 8.4 shows the seven hexagonal covers where towers can be installed. The purge tubes on the right are removed before closure.

The OVC was the outer vacuum enclosure. It was a vertical right cylinder 22.5 in. diameter and 27 in. tall. The walls were 1/8 in. thick and the top and bottom lids were 5/8 in. thick. The insulating vacuum is continuous with the outer vacuum of the dilution refrigerator. The removable lid and stem connections were sealed with Viton O-rings. The bottom of the OVC rested on the internal polyethylene shielding.

The Shield can operated at 80 K. It was wrapped with multilayer insulation (MLI) to reduce thermal radiation from the OVC. The inside was gold plated over nickel flash to reduce thermal emissivity. It is conduction cooled through the C-stem back to the liquid nitrogen reservoir in the dilution refrigerator. The Shield Can is not vacuum leak tight, but does provide a complete thermal radiation barrier and support heat intercept for the IVC. The shield can was 20 in. in diameter and 24.4 in. tall.

The IVC can operated at 5 K. The inside and outside were gold plated to reduce thermal emissivity. It was conduction cooled through the C-stem back to the liquid helium bath in the dilution refrigerator. The IVC separated the inner and outer vacuums. The inner vacuum was continuous with the dilution refrigerator inner vacuum and with the electronic feedthrough box. This can contained two lids; and inner lid that provided termination and final heat sink for the signal cables, and an outer lid that served as a vacuum tight cover.

All seals inside the background shielding were made with gaskets of annealed C101 copper sheets pressed between two bull nose raised surfaces with a 0.044 in. radius. Alignment pins on the outer lid and the stem connections ensured that opposing bull nose ridges matched each other. Fastening screws applied enough force to yield the annealed gaskets. Figure 8.5 shows the bull nose design. Seals outside the background shielding were made with indium wire. The IVC was designed to withstand full vacuum or atmospheric pressure with or without vacuum in the OVC.

The main purpose of the IVC was for detector cool down. The inner layers are thermally isolated from one another both in the cans and the dilution refrigerator, good for normal operation but not for cool down. By adding helium gas at a pressure of 0.1 atmosphere inside the IVC, inner cans and detectors become thermally well connected. This gas remains in place during cool down until a temperature of about 7 K is reached, at which time it is evacuated. The OVC remains fully evacuated during cool down, maintaining the MLI performance.

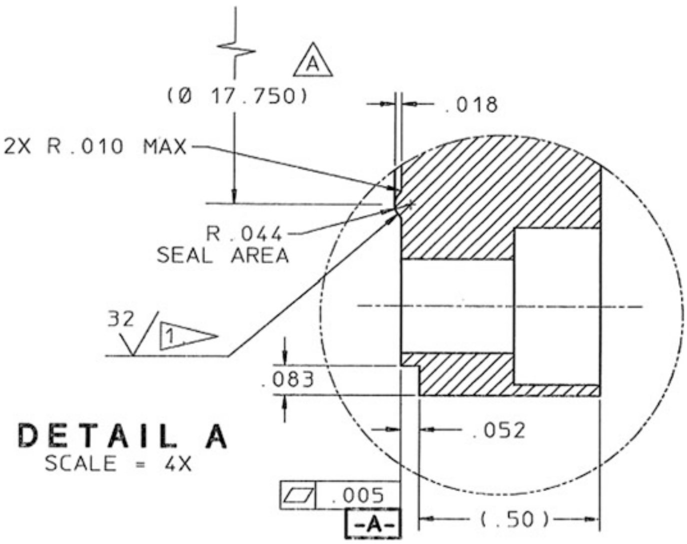

Fig. 8.5 IVC seal design [11]

The Still can operated around 1 K. It was conduction cooled through the C-stem back to the dilution refrigerator's still stage. The Still can is not vacuum tight but does provide a thermal radiation barrier and heat intercept for the CP layer supports. The Still can is 16 in. diameter and 16 in. tall.

The CP can operated around 230 mK. It was conduction cooled through the C-stem back to the dilution refrigerator. The refrigerator connection was at a midpoint in the counterflow heat exchangers between the still and the mixing chamber. The CP can is not vacuum tight be does provide a heat intercept for the MC layer supports. The CP can is 14.3 in. diameter and 14.3 in. tall.

The MC is the innermost can and operated at 56 mK. It was conduction cooled through the C-stem back to the dilution refrigerator. This can was connected to the dilution refrigerator mixing chamber. It provided heat sinking for the detectors and supported the weight of the detectors and towers. Both of those functions were carried out through the lid, which was made of a flat plate with hexagonal openings. It was 12 in. diameter and 12 in. tall.

8.5.2 Suspension

The OVC can rested on the inner polyethylene shielding. The five inner cans were each suspended on aramid fiber strings looped over pulleys attached to the top of the next warmer can. Aramid ropes have very low thermal conductivity and low elasticity. The loops were attached at the top of the warmer can and the bottom of the colder can. The adjustment screws and rope arrangement for each can allowed height adjustment and leveling. The innermost MC can had three equally spaced suspension loops. A flexible cable in the C-stem allowed some motion between the fridge and the MC can. The next four inner loops were suspended on two loops diametrically opposed and perpendicular to the stem attachment. For these four cans the dilution refrigerator provided the third support point, balancing the cans. The side loops were carefully adjusted to match the tail flanges with the fridge bottom flanges.

8.5.3 C-Stems and Tails

The Fridge Tails and Cold Stem connected the dilution refrigerator with the ICEBOX. All six layers were continuous to the associated layers in the fridge, which provided all cooling.

Changing the stem direction from horizontal to vertical was a challenging task. This was accomplished with five nested tees and a bolted conduction rod connected to the bottom of the fridge. The fridge bottom connection is shown in Fig. 8.6.

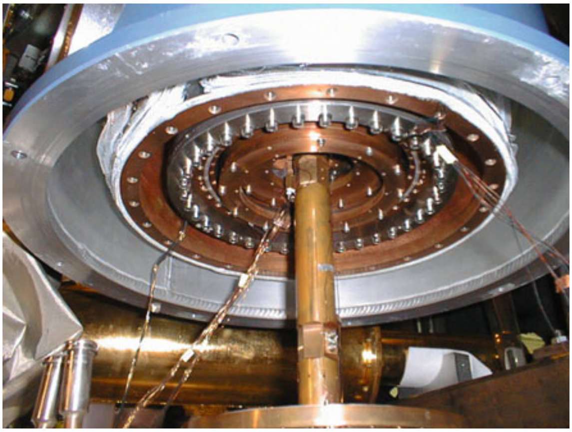

Fig. 8.6 Bottom of dilution refrigerator [17]

8.6 E-Stem

The E-stem carried the signal cables from the top of the towers at 5 K to the room temperature feedthrough box. It extended the OVC which terminated at the outer end of the E-stem. The LN shield layer extended though the E-stem and terminated with a stainless steel bellows at the outer end. A signal cable heat intercept at 90 K was located at the outer end of the stem. The LN layer in the E-stem was gold plated for low emissivity.

The IVC also extended through the E-stem. It carried the signal cables with a 5 K heat intercept near the can end. The IVC vacuum extends through the E-stem and into the feedthrough box. The E-stem inside diameter was 2 in., large enough to handle the maximum number of signal cables and their heat sinks at 90 and 5 K. The inner, narrow portion of the E-stem passed through the gamma and neutron shielding. The larger, outer portion contained the bellows isolating the inner and outer vacuums and temperature layers.

8.6.1 Thermal Contraction

Aramid loops have a slight thermal expansion, but the copper cans shorten when cooled, effectively lowering each can relative to the OVC. And since the dilution refrigerator is internally suspended from the top, it raises the balancing supports when cold. Therefore the ICEBOX cans lower and tilt when cooled down. The nested stems fit very closely together and the heights were adjusted so that they would have the maximum clearance cold. This meant that some adjacent stems were touching at room temperature but separated during cool down. Flexible connections in the E-stem allowed the LN and IVC to bend neat the can slightly during cool down, and bellows at the outer end of the E-stem allowed contraction along the stems.

8.6.2 Materials, Radiopurity

The materials used in the ICEBOX were strictly controlled to reduce the risk of radioactive contamination. Many common materials used in cryostat fabrication contain small amounts of radioactive contaminants or isotopes, so consequently they could not be used. Copper alloy C101 was the primary cryostat material, the highest purity available commercially. Although higher purity copper can be made with lower oxygen content, it is not available in the required quantities. One of the fabrication goals was to minimize cosmogenic activation which occurs after electrostatic refining when the material is above ground. The material was purchased with the most recent refining date reasonably achievable. Then the copper was stored underground when not needed and brought to the surface for fabrication.

Fasteners were primarily socket head brass screws and brass alignment pins. These were custom made from free machining brass rods, again for material control. Temperature sensors were RuO_2 or platinum resistors, connected with phosphor bronze wiring, and either polyimide or Teflon wire insulation. Millmax pins and counted solder were used to terminate sensor wiring inside the ICEBOX.

Samples of all materials used were tested in a low background radiation counter before fabrication.

8.6.3 Fabrication

All machining was done with carefully cleaned work areas and new tools to reduce the risk of embedding foreign material. All Icebox welding was done with electron beam process so that no filler metal was needed. Following fabrication, the copper assemblies were cleaned in an acid bath to reduce radon daughters and other surface contaminants acquired during fabrication, rinsed with water and wrapped for cleanliness. Gold plating over nickel flash will be applied where needed for thermal emissivity. Following cleaning all handling was done with powder free clean room gloves. One enhancement that could have improved thermal conductance of the joints would have been gold plating of all joint surfaces.

Tapped holes were made with rolling taps. This reduces the risk of embedded tool particles from cutting taps. It has the additional benefit of work hardening the threads, increasing their strength.

The conductive tubes and rods in the C-stem were annealed to achieve high thermal conductivity. The annealing was held at 410 °C in vacuum for one hour and slowly cooled. This process followed a procedure described by Fickett [6] to achieve a high RRR in oxygen free copper (see Fig. 8.7).

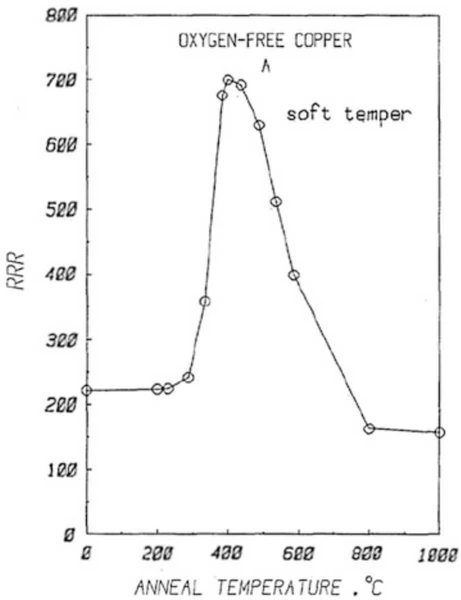

Fig. 8.7 Anneal temperature for maximum RRR [6]

8.6.4 Underground Assembly

All parts were again wiped with alcohol before introduction into the clean room. All handling was done with using cleanroom gloves, lint free cleanroom wipes, cleaned fixtures and tools. The nesting of the cans, stems and dilution refrigerator attachment required a specific assembly sequence. Icebox cans and the associated C-stem and E-stem were assembled from the outside in. The OVC, outer vacuum container, was mounted and leveled on the lead shielding. The OVC C-stem and E-stem were attached, temporary covers applied and the joints were leak tested. All seals on the OVC were made with Viton O-rings.

The gold plated joints were cleaned with alcohol wipes. All bare copper joints were prepared for assembly by scrubbing the stem connections with counted Scotch Brite™ and cleaning with alcohol wipes. This removed surface corrosion immediately before assembly and the wipes removed any particles from the cleaning. The aramid loops were attached to the can and a lifting fixture was used to lower the LN can into the OVC can. The C-stem and E-stem were inserted into the OVC stems and connected from the inside. All screws were tightened following a pattern using a torque screwdriver. All screws were assembled dry and tightened to 90 % of their breaking torque. Tests had shown that the breaking torque was repeatable for both brass and stainless machine screws. And even stainless screws would break before damaging the rolled threads. Stainless screws were only used outside the lead and polyethylene shielding.

8.7 Thermal Model

Thermal modeling of detector and cryostat elements is crucial, but with the paucity of consistent low temperature data and joint conductance information can be a challenge. Material properties above 4 K are readily available from NIST Cryogenic Properties Database (NIST) and the older Brookhaven National Laboratory selected cryogenic data notebook [9].

8.7.1 *Thermal Conductivity*

Conduction through solids below 4 K requires data from various sources. For CDMS the two materials of consequence were Kevlar (aramid fiber) and copper. Plotting test results from several reports shown in Fig. 8.8, the NIST (NIST) and Ventura [26] data appeared to be consistent over a wide temperature range. And with higher conductivity than other reports they are the appropriate choices for conservative design.

Copper thermal conductivity at low temperature is highly dependent on purity, heat treatment and operating temperature. Extremely high thermal conductivity can be obtained with 5 or 6 nines copper treated to remove oxygen and annealed. However this is not practical for the large conduction tubes needed for a cryostat such as the CDMS Icebox.

When large conductors were needed, the highest purity commercial alloy was used. C101, aka oxygen free copper, is readily available in industrial sized sheets, tubes and bars. Annealed copper following the procedure recommended by Fickett can achieve reasonably high conductivity (see Fig. 8.7) [6]. Risegari reported test data for annealed oxygen free copper between 30 and 150 mK. Risegari [19] Extrapolating the NIST data for RRR = 150 below 4 K, assuming a linear relationship with temp between 5 and 10 K does not line up very well with the Risegari equation.

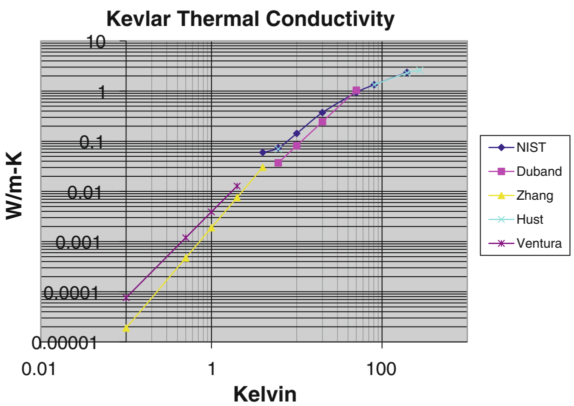

Fig. 8.8 Low temperature thermal conductivity of aramid fibers (NIST) [5, 8, 12, 14, 26]

8.7.2 Joint Conductance

Bolted joints are required for assembly of most cryostats but can add a significant conductance resistance in the millikelvin range. Copper surfaces readily corrode, and even a small amount of corrosion can impose a significant decrease in joint conductance. For CDMS the major can and stem connections were rigorously scrubbed and cleaned immediately before assembly and performed reasonably well. But it early operation it was clear that joints assembled with less rigor performed poorly.

For SuperCDMS a study of over twenty-five publications regarding joint thermal conductance was performed. Unfortunately, a large majority of the results were not directly applicable to the SuperCDMS experiment because:

- Joint conductance results relied on electrical resistivity measurements and the use of the Wiedemann-Franz Law, whose use across joints was questioned in Nilles [13] and Didschuns [4]
- Joint clamping force was unknown
- Use of foreign materials such as grease between the joint surfaces. Use of interfacial materials is to be avoided in SuperCDMS to maintain experimental radiopurity and general cleanliness.

Upon review of the pertinent publications; Nilles [13], Kittel [10], Didschuns [4], and Woodcraft [29], a design basis was chosen. Parameters for surface finish, plating and clamping force were chosen that could be readily achieved in a project with dozens of conductive joints and hundreds of bolts. Subsequent tests from 60 mK to 14 K in three refrigerators confirmed the validity of this design equation (Eq. 8.1) [24]. The design basis and test results are shown in Fig. 8.9.

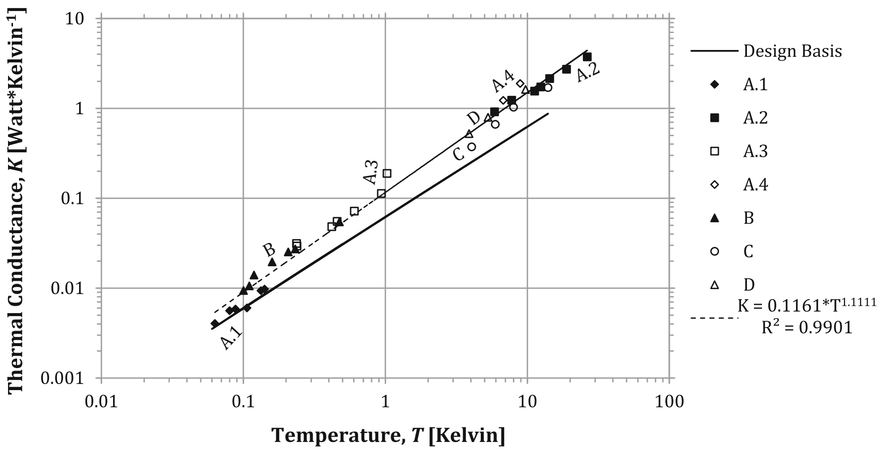

Fig. 8.9 Gold plated copper joint conductance, design basis and test results [24]

$$K = 0.0624\,T - 0.00023 \tag{8.1}$$

where:
K Joint Conductance [W/K]
T Temperature [K]

8.8 Heat Load

Heat load calculations between stages were dominated by the detector tower structure, signal wiring and wiring insulation. The thermal conductivity varies widely as a function of temperature and a list of relevant papers for design below 1 K may be most useful for the readers

Nb-Ti, [15]
SS316, [1]
Kapton HN, [2]
Kapton HN, [20]
Graphite CFRP, [21]
Ti15333, [27]
POCO AXM-5Q, [28]
Manganin 4 % NI, [16]
Manganin 4 % Ni, [25].

8.9 Detector Signal Feedthrough

Vacuum feedthroughs are readily available for a variety of standard connector types. But when hundreds or thousands of signal wires must be accommodated the cost and space requirements of standard commercial feedthroughs can become prohibitive. A variety of designs have been used. CDMS constructed a feedthrough box from an ISO 400 six way cross. Two sides of the cross were fitted with vacuum pumping or cryostat connections. Eleven D50 connectors were welded into blank ISO 400 flanges on the other four sides. Strain relief was provided by brackets on the outside of the flanges.

Another approach is to press a printed circuit board between vacuum flanges. Connectors in the middle of the flange face inward. The signals traces can be single or multilayer and extend radially through the seal area of the board. Connectors outside the sealing surface face outward. Choice of seal type and number of board layers is mainly driven by space considerations.

8.10 Dilution Refrigerator

CDMS at Soudan used a wet (consumes liquid nitrogen and liquid helium cryogens) dilution fridge for cooling at all stages. Later modifications added cryocoolers for heat interception in the E-stem, nitrogen and helium re-condensation.

After startup difficulties were resolved, the dilution refrigerator was very reliable, with multiple runs over one year duration. The longest run was nineteen months, and these later runs were terminated for scheduling reasons, not fridge problems.

Several enhancements improved the reliability of the dilution refrigerator.

Larger, separate liquid nitrogen reservoirs replaced the original, single reservoir for the 80 K cold traps. This allowed temperature and level measurements to be added for automatic nitrogen filling. The original 80 K cold traps were used for the entire project.

The circulation pump with mechanical shaft seals was replaced with a magnetic drive pump, eliminating possible shaft seal leaks and seal maintenance. The pump oil was replaced with Fomblin SV for low oil vapor carry over. After draining and filling with the replacement oil, it was again drained and heated in a beaker, from which the original oil was skimmed off. The reliability of this pump has been excellent.

The dilution refrigerator was designed with a cold trap inserted in the bath. A second, external 4 K cold trap was added upstream and in series with the fridge internal cold trap. The external cold trap resided in a separate liquid helium bath. This external cold trap was initially filled manually from liquid helium supply dewars, but soon was equipped with a cryocooler helium liquefier, which maintained the liquid helium without further transfers. Very small amounts of helium leakage were made up with gas from high purity gas cylinders.

Regular, monthly cold trap regeneration was the key to long term reliable operation.

Most new experiments utilizing dilution refrigerators choose 'dry' models, which use a cryocooler(s) rather than liquids nitrogen and helium to provide the upper stage cooling. The dry fridges avoid the cost of liquid helium, the effort of dewar handling and liquid transfers. The latter is especially valuable underground or where access is limited.

8.11 Liquid Transfer Systems

The dilution refrigerator at Soudan was a wet dilution refrigerator of a type typically installed at the time. With the heat loads from the Icebox, it required daily transfers of liquids nitrogen and helium. Common practice for many refrigerators at that time was to manually transfer cryogenic liquids. But the Soudan underground location with limited access required an automated solution. It was also important to use liquid cryogens efficiently to minimize cost and effort to transport dewars underground.

Dual 160 L nitrogen supply dewars were connected to a vacuum insulated manifold outside the RF room. During transfers, liquid was drawn from the lead supply dewar. If it ran dry during the course of a transfer the backup dewar would complete the transfer. The backup would then become the lead for the next transfer, allowing time for the empty to be replaced. Inventory was tracked by scales located under each dewar.

160 L nitrogen dewars normally have pressure building regulators and vent valves. But those regulators often provide imprecise control and/or leak through wasting liquid nitrogen. More precise pressure control contributes to faster transfers without risking an overpressure in the fridge. When the liquid withdrawal line was connected to a dewar, a gas management line was also connected. The gas management line was equipped with pressure measurement, gas makeup and vent valves. During standby the PLC operated solenoid valve vented nitrogen if the pressure was too high. During transfers pressure was maintained with another solenoid valve supplied from high pressure cylinders.

When a transfer was initiated, a gas line pressurized the supply dewar and a cool down valve opened. When the transfer line was cooled down, the valve(s) filling the dilution refrigerator or 80 K cold traps opened and the cool down valve closed. A valve sequence after filling was used to ensure transfer efficiency and prevent trapped volumes in the piping.

Dual 350 L liquid helium dewars were connected to a vacuum insulated manifold outside the RF room. This was the largest helium dewar size that would fit on the mine shaft elevator. Dewar selection, pressurization, inventory and transfer procedures were similar to the nitrogen system. The helium transfer valves were close to the dilution refrigerator inside the RF room and therefore were pneumatically controlled from outside.

To minimize detector down time the helium and nitrogen transfers could be automatically synchronized. Transfers could be initiated by time-of-day, by low nitrogen or helium level in the fridge, or manually.

8.12 Liquefier Addition

The initial operation of the dilution refrigerator required daily transfers of both liquids. During transfers the physics data taking was stopped, losing an hour each day of experiment run time. Transporting dewars underground was typically done weekly, taking several hours with the elevator doors removed, technicians at the top and the bottom of the shaft. Normal deliveries to the remote site were weekly although special shipments could be made.

As the commercial development of cryocoolers progressed it became feasible to add liquefiers to the system. For this project all cryogenic equipment had to be installed outside the RF room, complicating the installation.

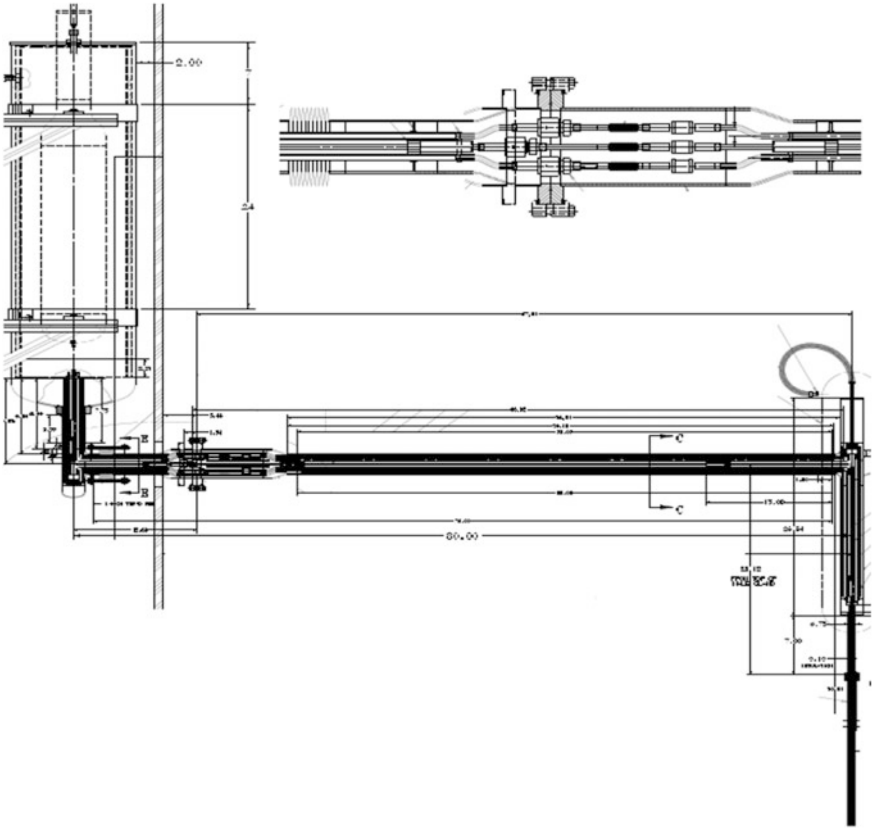

Fig. 8.10 Low heat load transfer line between liquefiers and dilution refrigerator [23]

A transfer line was needed between the top of the dilution refrigerator to a bayonet box outside the RF room. The RF wall penetration, the thermo siphon design and the low heat load requirements prevented the use of a U-tube separation. Instead the transfer line had an S shape from the liquefier connection to the fridge. The line included an actively cooled 78 K heat shield, which extended into the male bayonet into the fridge. Assembly was made at the midpoint with a retractable bellows and VCR connections. A spare port on top of the fridge was modified to accept a custom male bayonet. The transfer line total length was 100 in. The transfer line is shown in Fig. 8.10.

The nitrogen liquefier was a GM type cryocooler was fitted with a desuperheating/condensing heat exchanger and installed in a custom vacuum vessel. An electric heater embedded in the heat exchanger was used to remove excess refrigeration capacity. The cryocooler vacuum vessel was mounted on rubber isolators to reduce the vibration forces carried into the support structure. During holding operation the heater was controlled by the nitrogen reservoir pressure.

Small losses were replenished by automatically adding gas from the liquid nitrogen supply dewars. The nitrogen liquefier system was very reliable, typically operating with about 120 W of excess capacity.

The dilution refrigerator was designed to use the helium boil off gas to intercept heat through the bath neck. Superheating helium gas also provided some cooling the nitrogen reservoir. Consequently adding a helium condenser would not have been feasible. The steady state heat load from the Icebox and dilution refrigerator consumed about 20 liquid liters/day. This was marginally within the capacity of two Cryomech PT-415 liquefiers, but with the transfer line heat load they were not quite enough capacity. With a third liquefier the cooling capacity was more than enough for the system. Electric heaters in the liquefiers were used to compensate for the excess cooling power. Heater power control was based on helium bath pressure.

Liquid helium from the refrigerator bath fed the 1 K pot. Helium from the pot was not recovered, so that loss plus leakage required regular makeup. Makeup gas was taken from helium supply dewar boil off. Although this system could run for months at a time, eventually trace amounts of contaminants would build up in the transfer line and stop the thermo siphon. Recovery involved disassembly of the transfer line, removal from the dilution fridge and a warmup. This could normally be done in one day without disturbing the fridge base temperature operation. A description of the liquefier system was published in Advances in Cryogenic Engineering [23]. The source of contamination was not known, but an easier means of removing and warming up the transfer line would have been an improvement.

8.13 External Cold Trap

Small amounts of contaminants in the mix would gradually plug the internal cold trap. To improve the operating reliability a 4 K external cold trap was installed downstream of the 80 K traps. This trap dewar was initially filled with liquid helium transfers, but was fitted with a helium liquefier. The liquefier could handle the total heat load and slowly fill the cold trap dewar from high pressure gas cylinders. The liquefier heater was controlled by dewar pressure, which could also add or vent helium gas when required. This system was very reliable.

8.14 E-Stem Cryocooler

During initial operation with fewer detectors it became apparent that the E-stem conduction and heat sinks would not be adequate for more detectors. A two stage SHI cryocooler was fitted to the E-stem to intercept signal cable heat. To minimize vibration it was independently suspended and equipped with vibration isolation. Extra thin, edge welded bellows isolated the vacuum shell from the ICEBOX. Braided cables isolated the first stage and bundled fine wires isolated the second stage. Even with this support and isolation system noise from the GM cryocooler was troublesome for the detectors.

8.15 Insulating Vacuum

The insulating vacuum inside the system must be good to reduce the heat load, and it is very important to limit the helium that could enter the IVC through leaks or permeation. Dry roughing and turbo molecular pumps were used to evacuate the ICEBOX. They were equipped with automatic valves that close in case of power loss or pump failure.

Standard dilution refrigerators have evacuation tubes sized for typical small applications. These tubes are not large enough to reasonably evacuate the larger volumes in the ICEBOX. To improve the pumping speed for the OVC an adaptor and custom radiation baffle was added to the bottom of the fridge tails. It was the equivalent of a 3 in. diameter pumping line and had no noticeable effect on the helium bath heat load. Pumping lines were added to the signal feedthrough box for the IVC.

8.16 Automation and Control

The cryogenic system was monitored and controlled with an industrial PLC (programmable logic controller) and HMI (human machine interface). They were both current industry products during installation and most of the operating period. The automation and control system was backup up by UPS (uninterruptable power supply) and standby generator.

This system provided local and remote monitoring, local and remote control, historical cryogenic data collection and fully automated operation. Automatic cryogen transfers, recovery from power outages, liquefier heater control and so on were fully automated.

The millikelvin systems at Soudan did not have temperature control, generally running as cold as possible. Helium and nitrogen liquefiers on systems on the other hand must be controlled to avoid subatmospheric pressure and freezing the nitrogen. Resistance heaters in the liquefiers, with zero crossing SCR's were controlled using conventional PID loops with cryostat pressure as the process variables. Pressure measurement is sensitive and small measurement errors will not cause the system to operate below atmospheric pressure.

Much more complicated are the sequences, abnormal procedures and alarms. Some examples from Soudan included conditional alarms, automatic set point adjustment, restart sequences after power outages, backup cooling water start, cryogen transfer sequences, etc. A modern industrial PLC with IEC 61131-3 programming languages can be configured to carry out these automation tasks in graphical form, without resorting to complicated scripts.

8.17 Cryogenic Operation

The remote location made commissioning and early operation more difficult. During the early years there were several operational problems that required the fridge to be removed from the ICEBOX. Once settled, however the fridge operation became very reliable. There were multiple runs at 56 mK over a year in length with the longest at 19 months. These runs were usually stopped for reasons unrelated to the dilution refrigerator. The dilution refrigerator and ICEBOX were in service for twelve years.

8.18 Lessons Learned

8.18.1 Cryogenic System Assembly and Testing

After successful shop tests there was an assumption that after shipping and reassembly the dilution refrigerator would operate successfully. But several cool down attempts were required to commission the fridge and support equipment, repair leaks and damage, etc. Repairs were made more difficult by the underground, remote location. Future experiments in remote locations should consider a full operational test of all cryogenic systems at a convenient location.

8.18.2 Wiring

Thermometer and touch sensor wiring was routed through the dilution refrigerator, which was equipped with cryogenic rated micro D connectors and heat sinks at each stage. While this was thermally and functionally successful, routing these cables along the narrow C-stem was difficult. A better solution would be to include these signal wires along with the detector signal cables.

8.18.3 Mixture Purification

The standard fridge came equipped with a 4 K cold trap immersed in the helium bath. Anything that gets past this trap can cause plugging at the condenser impedance and requires a subsequent warmup. The installation of an additional 4 K cold trap reduces the risk of trap plugging during long runs.

8.18.4 Micro Vibrations

The initial installation did not utilize cryocoolers, but their installation caused noise pickup in the experiment detectors. Even with typical mechanical isolation methods the transmitted noise was significant. It is thought that when excited, the SNOBOX cans vibrated at their natural frequencies, then transmitted the vibration through the suspension to the adjacent inner cans. Transmissibility could have been reduced by installing springs on the hanger supports, especially at higher frequencies. Future projects should consider transmissibility of impulse from outside the cryostat or even the negative spring constant concept.

8.18.5 Can Supports

The aramid fiber loops effectively supported the cans with low heat load. However they were constructed of sixteen loops each. The inelasticity of Kevlar string required the loops to be identical in length and consequently a more difficult assembly procedure. More recent projects have used a single loop made of a larger braided rope, simplifying assembly.

8.18.6 Inner Vacuum, Yes or No?

CDMS II at Soudan has a sealed Inner Vacuum Chamber (IVC), a separate vacuum space from the Outer Vacuum system. During cool down, above 10 K, this IVC space is filled with a small amount of helium exchange gas to thermally link the inner cans to the IVC. While successful, this approach requires a leak-tight chamber at 5 K and the ability to evacuate helium gas at 10 K before proceeding below 10 K. For CDMS the cooldown was accomplished with several steps: Introduce liquid nitrogen into the helium bath and nitrogen shield reservoir, purge all liquid and evacuate the nitrogen from the helium bath, slowly introduce liquid helium, evacuate the exchange gas. Then start the dilution refrigerator.

Since a dry fridge does not have liquid nitrogen or helium reservoirs, and since the typical two stage cryocooler does not have enough capacity to reasonably cool down a large cryostat, it is more appropriate to dispense with the IVC and use cooling tubes fixed to each stage for cool down. For a system of this type heat exchangers attached to each stage would be linked in series using stainless steel bellows. Helium gas coolant is circulated through these heat exchangers and out to cryocoolers. This allows a large single stage cryocooler to start the cooldown at room temperature, and a two stage cryocooler can take over below 40 K. Once the layers are cold the helium gas will be evacuated from this cool down circuit. The long conduction path provided by the bellows shape and the poor thermal conductivity of stainless steel limits the heat load between layers to an acceptable value once the helium is evacuated form this circuit.

References

1. M. Barruci, Measurement of thermal conductivity of the supports of CUORE cryostat. Cryogenics **48**, 166–168 (2008)
2. M. Barucci, Low temperature thermal conductivity of Kapton and Upilex. Cryogenics **40**, 145–147 (2000)
3. CDMS at Fermilab (2003). Retrieved from http://ppd.fnal.gov/experiments/cdms/. http://titus.stanford.edu/public/photos/cdms_icebox.jpg
4. I. Didschuns, Thermal conductance measurements of bolted copper to copper joints at sub-Kelvin temperatures. Cryogenics **44**, 293–299 (1992)
5. L. Duband, Thermal isolation of large loads at low temperature using Kevlar rope. Cryogenics, 643–647 (1993)
6. F. Fickett, Oxygen free copper at 4 K: resistance and magnetoresistance. IEEE Trans. Magn. **19**(3) (1993)
7. G. Frossati, Experimental techniques: methods for cooling below 300 mK. J. Low Temp. Phys. **87**(3/4) (1992)
8. J. Hust, Low-temperature thermal conductivity of two fibre-epoxy composites. Cryogenics, 126–128 (1975)
9. J. Jensen, *Brookhaven National Laboratory Selected Cryogenic Data Notebook* (1980)
10. P. Kittel, Thermal conductance of gold plated metallic contacts at liquid helium temperatures. Adv. Cryog. Eng. **37**, 241–248 (1992)
11. LBL. (1993). IVC Seal Design
12. E. Marquardt, *Cryogenic Material Properties Database*. 11th International Cryocooler Conference (Keystone, Co, 2000)
13. M. Nilles, Effects of oxidation and roughness on Cu contact resistance from 4 to 290 K. Adv. Cryog. Eng. **34** (1987)
14. NIST (n.d.), *NIST Materials Measurement Lab, Cryogenic Technologies Group, Material Properties*. Retrieved from http://cryogenics.nist.gov/MPropsMAY/materialproperties.htm
15. J. Olson, Thermal conductivity of some common cryostat materials between 0.05 and 2 K. Cryogenics **33** (1993)
16. I. Peroni, Thermal conductivity of manganin below 1 K. Nucl. Phys. B **78**, 573–575 (1999)
17. D. Bauer, PAC Meeting, March 30, (2007). Retrieved from https://www.fnal.gov/directorate/program_planning/March2007PACPublic/BauerPAC03_07.pdf
18. M.D. Resources, *Lake Vermilion-Soudan Underground Mine State Park* (2015). Retrieved from http://www.dnr.state.mn.us/state_parks/lake_vermilion_soudan/index.html
19. V. Risegari, *The Art of Cryogenics: Low Temperature Experimental Techniques* (Elsevier, Oxford, UK, 2008)
20. D. Rule, Thermal conductivity of polypyromellitimide film with alumina filler from 4.2 to 300 K. Cryogenics **36**, 283–290 (1996)
21. M. Runyan, Thermal conductivity of thermally-isolating polymeric and composite structural support materials between 0.3 and 4 K. Cryogenics **48**, 448–454 (2008)
22. J. Sander, *Fermilab Today* (2015). Retrieved from http://www.fnal.gov/pub/today/archive/archive_2015/today15-02-06.html

23. R. Schmitt, Application of cryocoolers to a vintage dilution refrigerator. Adv. Cryog. Eng. (2011)
24. R. Schmitt, Thermal conductance measurements of bolted copper joints for SuperCDMS. Cryogenics (2015)
25. Y. Touloukian, *Thermophysical Properties of Matter: Thermal Conductivity, Metallic Elements and Alloys*, vol. 1 (IFI/Plenum, New York, 1970)
26. G. Ventura, Low temperature conductivity of Kevlar. Crygenics, 489–491 (2000)
27. P. Wikus, The electrical resistivity and thermal conductivity of Ti 15V–3Cr–3Sn–3Al at cryogenic temperatures. Cryogenics **51**, 41–44 (2011)
28. A. Woodcraft, Thermal conductivity measurements of pitch-bonded graphite at millikelvin temperatures. Cryogenics **49**, 159–164 (2009a)
29. A. Woodcraft, Thermal design and performance of the SCUBA-2 instrument 1-K and mK systems. Cryogenics **49**, 504–513 (2009b)