Salt Pill Design and Fabrication for Adiabatic Demagnetization Refrigerators

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ABSTRACT

The performance of an adiabatic demagnetization refrigerator (ADR) is critically dependent on the design and construction of the salt pills that produce cooling. In most cases, the primary goal is to obtain the largest cooling capacity at the low temperature end of the operating range. The realizable cooling capacity depends on a number of factors, including refrigerant mass, and how efficiently it absorbs heat from the various instrument loads. The design and optimization of "salt pills" for ADR systems depend not only on the mechanical, chemical and thermal properties of the refrigerant, but also on the range of heat fluxes that the salt pill must accommodate. Despite the fairly wide variety of refrigerants available, those used at very low temperature tend to be hydrated salts that require a dedicated thermal bus and must be hermetically sealed, while those used at higher temperature – greater than about 0.5 K – tend to be single- or poly-crystals that have much simpler requirements for thermal and mechanical packaging. This paper presents a summary of strategies and techniques for designing, optimizing and fabricating salt pills for both low- and mid-temperature applications.

INTRODUCTION

In the early 1980s, ADRs began a period of re-development that paralleled the development of low temperature detectors for space missions. Initial efforts focused only on ADRs for specific instruments, and specific cooling requirements. However, their solid-state nature, robust magnetic cycle, and ease of integration with cryogenic systems also made them convenient coolers for laboratory facilities, both in support of instrument development and as multi-purpose systems. As detector technologies improved and their cooling requirements became more challenging, ADRs began a shift toward multi-staging that enabled wider operating range, higher cooling powers, and cryogen-free operation. The development of multi-stage systems required significant changes to the salt pills that contain the magnetic refrigerant – not only in the heat loads they can accommodate, but also the temperatures at which they operate and the materials used.

Early ADRs[1, 2] consisted of single stages – salt pill, magnet and heat switch – typically designed to operate at very low temperature (below 100 mK) using a superfluid helium bath (at ~1.5 K) as a heat sink. Cooling is based on the magnetocaloric effect driven by the interaction of a system of electronic spins (as opposed to nuclear spins in nuclear demagnetization systems) with an external magnetic field. In Figure 1, the specific entropy is shown for an idealized ADR cycle, which consists of 4 basic steps. Starting from low temperature and (usually) zero magnetic field, the stage is adiabatically magnetized by steadily increasing the magnet's current. Increasing field has the effect of warming the salt pill, and when its temperature exceeds that of the heat sink, the heat switch is turned on. Continued magnetization then occurs isothermally as heat from the salt pill flows to the sink. At full field, the heat switch is turned off, and the stage is adiabatically demagnetized to cool the salt pill. When the salt pill, or attached load, reaches its operating point, the magnet current is typically regulated in a feedback loop to control the salt pill (or load) temperature. The stage undergoes slow isothermal demagnetization to stabilize its temperature as it absorbs heat. When the field is reduced to zero, the process is repeated.

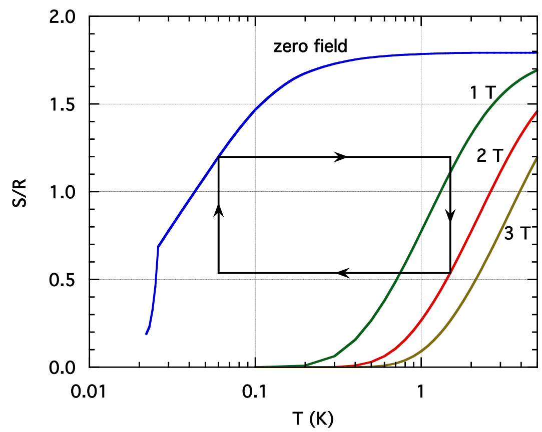


Figure 1. Entropy diagram of a single-stage ADR containing FAA salt, operating at 60 mK and using a 1.5 K heat sink. (Entropies from Vilches & Wheatley 1966[3]).

At very low temperature, the only available refrigerants are hydrated salts, which must be encapsulated in a hermetic container to prevent dehydration. Because of the salts' high thermal boundary resistance, a high surface area thermal bus in included to enhance thermal contact. Various thermal bus concepts have been used, but the most efficient are those which used a grid of wires – most often gold because of chemical compatibility issues – onto which the salt is grown from solution.

For single-stage, single-shot ADRs, the need to store heat for long periods means that even kg masses of refrigerant have limited cooling power – at most a few microwatts. The thermal bus is therefore optimized for low heat fluxes, and can be very small, typically 2-3% of the salt pill volume.

In two-stage (and multi-stage) ADRs[4, 5], the coldest stage generally has lower a heat load than a comparable single-stage ADR would have, and the design of its salt pill is driven not only by the requirements for heat absorption at low temperature (T_{cold}), as for single-stage ADRs, but also for transferring heat between the two stages. The required heat flow rates can be scaled from the low temperature heat load, $\dot{Q}_{load}$:

$$\dot{Q}_{transfer} \sim \dot{Q}_{load} \frac{T_{transfer}}{T_{cold}} \frac{\Delta t_{hold}}{\Delta t_{transfer}} \quad (1)$$

where $T_{transfer}$ is the temperature where heat is transferred, Δt_{hold} is the ADR's hold time, and $\Delta t_{transfer}$ is the time allotted for the transfer. The ratio of temperatures is typically in the range of 10-20, and the ratio of times can be another factor of 50-100. For a heat load of microwatts, the transfer rate must be in the milliwatt range. Typically $T_{transfer}$ is above ~1 K, where thermal boundary resistances are small, so the emphasis for salt pill design is to ensure the thermal conductance along the thermal bus is large compared to that of the heat switch.

Continuous ADRs[6] (CADR) can impose even more challenging requirements for salt pill design, since comparatively large heat transfers must occur at even lower temperature where thermal boundary resistances are large. For an isothermal stage, $T_{transfer} \lesssim T_{cold}$, and $\Delta t_{transfer} \sim \Delta t_{hold}/3$. In a CADR capable of handling, say, 5 μW at 50 mK, heat must be transferred to the next stage at rates of at least 15 μW at ~45 mK. Since each stage in a CADR is smaller – by at least an order of magnitude – than is needed in a single-shot ADR to achieve the same cooling capacity, the challenge is to achieve much higher thermal conductance between the salt and the external interface in a smaller salt pill volume.

But regardless of the particular operating temperature, heat flow rates, etc., the goal of optimization is the same: to obtain the largest cooling capacity at temperature within the allocated salt pill volume. The process involves balancing the reduction in salt mass that the addition of a thermal bus entails, with the larger entropy capacity that is achieved through improved heat transfer efficiency. It also involves structuring the salt pill and thermal bus to avoid such problems as eddy current generation and weakly-coupled heat capacities, which can in turn dictate how the salt pill and thermal bus is manufactured. This paper presents a summary of techniques and strategies for the optimization and fabrication of salt pills in a range of ADR systems, from single-stage coolers to multi-stage CADRs.

Design and optimization of salt pills using hydrated salts

For temperatures below 0.5 K, the only choices for paramagnetic cooling are the various hydrated salts, where the primary tradeoff is between spin density and ordering temperature. Salts with higher magnetic moment density will have more cooling capacity for a given salt volume, but they will also have higher limits on the lowest temperatures that can be reached. The most common salts are manganese ammonium Tutton salt (MAS), ferric ammonium alum (FAA), potassium chrome alum (CPA), and cerium magnesium nitrate (CMN). The minimum practical temperatures for these are about 180 mK, 35 mK, 12 mK and 2 mK, respectively. Low temperature properties for the first three can be found in Vilches & Wheatley (1966) [3], and for CMN in Mess et al (1964) [7]. Secondary considerations such as corrosiveness, ease of crystal growth, and temperature stability (to withstand a modest bakeout of the cryostat containing them) may favor other choices. Hagmann et al (1994) [2] discuss some of these.

A complication of using these hydrated salts is that the equilibrium vapor pressure of the water of hydration is high at room temperature, and they decompose rapidly in a vacuum.

Epoxy seals eventually leak after many thermal cycles; the only satisfactory long-term solution is to enclose the salt pill in a hermetic container. This is usually a thin-wall cylinder of stainless steel, which resists corrosion, minimizes eddy current heating, and can readily be welded. Thermal bus penetrations usually must be brazed in place before the salt is grown, but with some design forethought it is relatively easy to weld the final closure without heating the salt significantly. As long as decomposition temperature limits are observed, salt pills of this construction have been used for many years and hundreds of thermal cycles without measureable degradation.

The thermal connection or "bus" from the cold plate to the salt requires careful attention along the entire path. Any significant thermal resistance along the bus results in operating the salt at a lower temperature than the load being cooled. This reduces performance two ways. First, more entropy is generated per unit heat absorption. Second, the salt pill's entropy capacity is reduced due to the rapidly falling value of the zero-field entropy with temperature. This is particularly true when the operating point is close to the ordering temperature, as can be seen in Figure 1. Demountable joints and thermal strap connections to the cold load are problematic. Many solutions are available in the literature[see for example 8, 9, 10], but all aspects of the external bus require careful quantitative analysis.

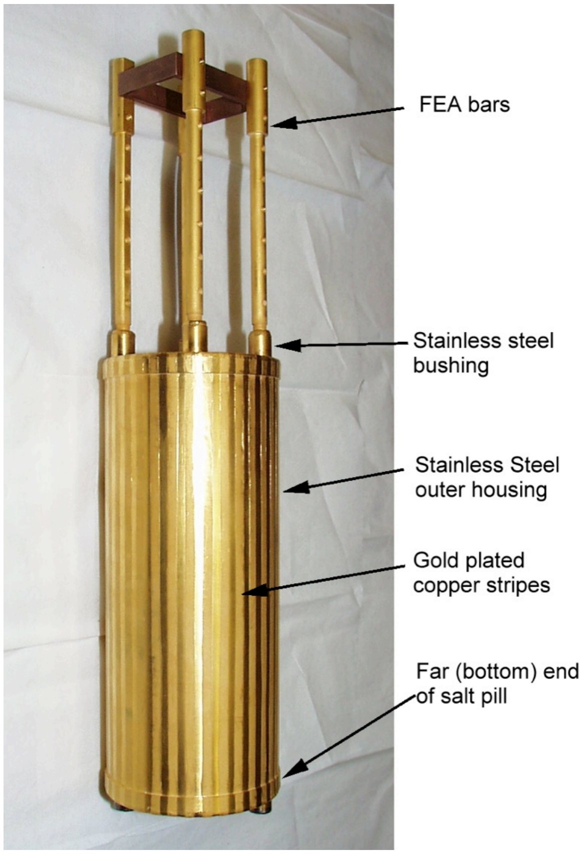
Thermal contact to the salt itself is usually made with a bundle of wires, often gold due to the corrosive nature of these salts. The bulk conductivity within each single crystal is much higher than that across the grain boundaries, so it is desirable to have the wire spacing somewhat smaller than the minimum size of the individual crystals in the usually polycrystalline pill. Most growth methods result in crystallites of roughly 5 mm diameters, so this sets the minimum density of wires required. The desired longitudinal conductivity determines the total cross section of the wires, and the requirement that Kapitza boundary resistance between the wire and the salt not add too much to the total wire resistance sets a lower limit on the surface area and therefore the size and number of wires required. Since the temperature scaling of the wire resistance (T^{-1}) and the boundary resistance (T^{-3}) are quite different, the optimization for low temperatures with a large number of small diameter wires to minimize boundary resistance, and for high temperatures where a smaller number of large wires will do, is quite different. And thermal contact with the salt is required at two different temperatures, above the bath during magnetization and somewhat below the cold plate temperature during operation.

These temperature are typically a factor of 30 or so apart for these salts, so the Kapitza resistance is unimportant during magnetization, but the desire for a short cooling time to obtain a high duty cycle (which can easily exceed 95%) requires a large total cross section that can result in an unmanageable number of wires if the small diameter required for the low temperature coupling is used. So it is often desirable to have two wire diameters in the thermal bus bundle, and there are a couple of factors that favor dividing them into two separate buses.

The first is that if the total heat input to the salt is dominated by the parasitic heat load from the suspension and off-state conduction of the heat switch, as is often the case, most of this heat can be diverted to the second (high-temperature) bus, and only the small fraction the heat that comes from the cold plate itself will flow through the low temperature bus. It should then be possible to make this temperature drop almost negligibly small with a modest bus that displaces very little salt (and has little weight). The high temperature bus will have a relatively large drop since it is not optimized for this temperature, but as long as the two buses are reasonably well isolated, it can be arranged that the majority of the parasitic heat load is carried by this secondary bus.

The other practical advantage of separate buses is reduction in eddy current heating and a more flexible mechanical arrangement of the thermal paths. The small diameter of the individual bus wires makes eddy currents induced within each wire negligible, but care must be taken not to make loops of this high-conductivity wire that enclose significant area. In practice, this means the bus needs to be open-ended: it is bundled together at one end to connect to a bus exiting the hermetic can, but shouldn't also be bundled at the other end for, say, convenient connection to the heat switch. With separate buses, the heat switch and cold plate buses can exit at opposite ends if this is desirable.

Figure 2 shows a large (1 kg) FAA salt pill in a welded stainless steel can. The four long thermal buses each have their own bundle of fine gold wires within the salt and provide parallel paths to the cold plate. The circular area between them is the end of a bundle of larger wires for the heat switch, which attaches at this point (and is the source of most of the 4 μW parasitic heat load). The longitudinal stripes on the sides of the pill are plated copper strips that help keep the long stainless steel casing in thermal equilibrium without adding significant eddy current heating. They also provide a path for the parasitic heat from the suspension to the heat switch bus. There is a thin overall gold plating for low emissivity.



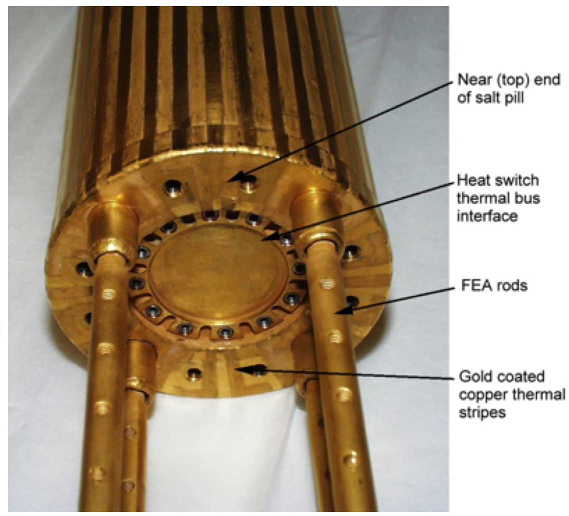


Figure 2. A 1 kg FAA salt pill in a welded stainless steel container. There are five independent thermal buses, but four of them are used in parallel for the cold plate connection.

The overall performance is affected by many details. Figure 3 shows the measured thermal conductance of each of the five buses in the salt pill of Figure 2, the first 4 of which connect to the Front End Assembly (FEA) containing the detectors. FEA #1 has almost the conductivity of the other three cold buses combined due to a higher annealing temperature used for this bundle, which was otherwise identical to FEA#2-4 (the heat switch bus has more and thicker wires). The measured conductivity difference is consistent with the difference in RRR obtained for this bundle: about 1100 compared to an average of 320 for the other three. If the better anneal had been used for all the wires, far fewer would have been needed, saving additional weight. Total non-salt weight is about 750 g, but could have been considerably less with fewer wires and a different design for the external buses.

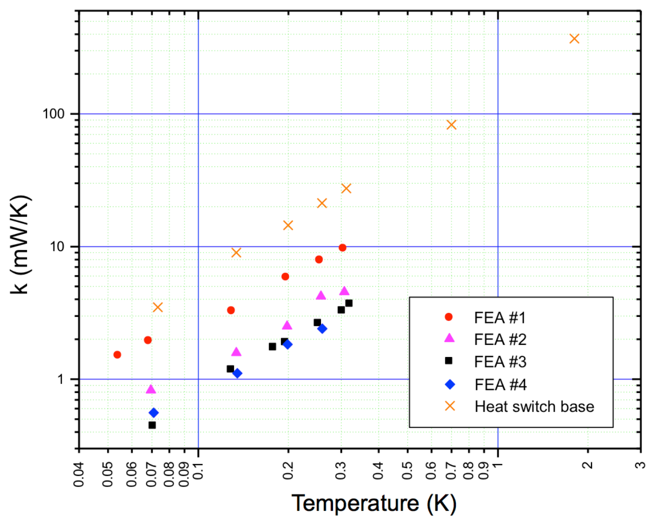


Figure 3. Measured thermal conductivity of the four thermal buses to the detector stage (FEA) and the heat switch bus.

Figure 4 shows a demagnetization cycle with eight thermometers monitoring different points in the salt pill structure, including the stainless steel case at the far end. Everything stays within a few millikelvins of equilibrium, ensuring a close to Carnot thermal efficiency. This salt pill will maintain 60 mK for over 35 hours and recycles in less than one hour with a gas-gap heat switch (>97% duty cycle), and holds 250 hours and recycles in 1.8 hours with a mechanical heat switch (>99%).

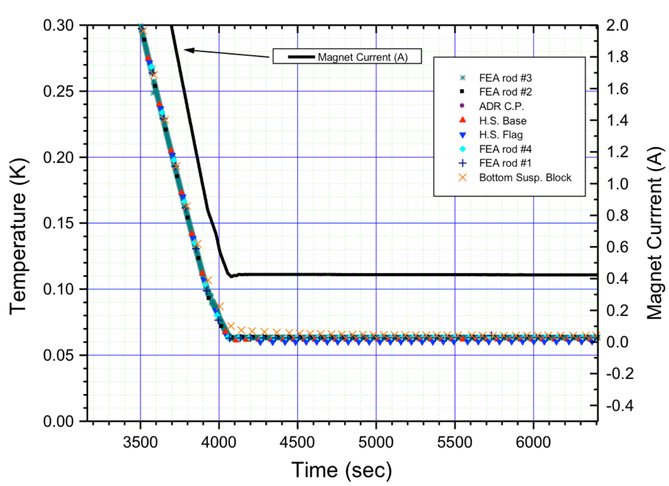


Figure 4. Demagnetization cycle showing several thermometers at various points on the salt pill structure.

Long-term stability of salt pills

There are several potential mechanisms for salt pills to degrade over time. Mismatches in coefficients of thermal contraction (CTE) could, over a number of thermal cycles, cause a failure of the bond between the salt and thermal bus. Unsaturated solution trapped in voids in the crystal structure could expand as the fluid freezes and dislodge the salt from the thermal bus. And breaches of the hermetic seal, possibly also due to CTE-induced stresses, could dehydrate the salt while in vacuum. Degradation from any of these causes would be readily apparent from increased gradients between the salt and thermal bus (if measured directly), from lower currents at the start of a hold time, or reduced hold times.

Anecdotal evidence suggests, however, that the bond between alum salts and metal (gold and copper) thermal buses is inherently stable. In addition, with careful attention to workmanship (fabrication and salt growing techniques), as well as rigorous testing for leaks prior to salt growth, the risks associated with the other failure mechanisms are minimal. Salt pills produced by the authors have undergone dozens of thermal cycles and years of use with no observable change in characteristics.

Suspension considerations: parasitic heat and resonant frequency

The salt pill and cold plate suspension system is an important contributor to the refrigerator performance. Britt and Richards (1981)[11] pointed out that as long as the heat load is dominated by the suspension and the total mass by the mass of the salt, the salt pill size can be scaled down and if the suspension cross section is scaled to keep the strength relative to salt mass constant, the resonant frequency and hold time will also be unchanged.

The figure of merit for the material used for the suspension is the ratio of its Young's modulus to thermal conductivity, rather than strength to thermal conductivity. As long as space is available, heat load can be reduced arbitrarily at a given strength by increasing the length of the suspension members at constant cross section. However, the maximum resonant frequency of a suspended pill at a given heat load is entirely determined by the material properties. Kevlar 49, with a tensile modulus of 112 GPa and a thermal conductivity of about $4 \times 10^{-3}\ T^{1.35}\ \mathrm{Wm^{-1}K^{-1}}$ in the 0.2-20K range is the best of the well-characterized materials.

Unfortunately, it is also difficult to use. For high efficiency, one has to use the untwisted yarn, and with only 2% elongation at the breaking point, it's not easy to ensure that there is tension in all of the very small individual fibers. For instance, running it around a pulley tends to leave the fibers on the inside untensioned. Keeping the yarn perfectly straight and gluing it into a longitudinal hole in a screw is very effective for small diameter yarns, but the strength doesn't scale well for heavier loads. On top of this, it gets longer when cooled, so a good mechanism is required to keep it in tension when everything else is contracting. It is tempting to encase it in an epoxy matrix to alleviate these problems and maintain uniform loading, but without a careful choice of materials and application the thermal conductivity of the epoxy is apt to exceed that of the Kevlar.

The suspension should be a kinematic design so that the resonant frequency can be maximized for a given thermal conductivity. This means that if the salt pill structure can be regarded as a rigid body, there should be exactly six supports of fixed length that determine its position. The geometry of these is arranged to make all the lowest principal resonant frequencies approximately equal. The mount points need to be very stiff to maintain the large spring constant of the Kevlar supports. Additional fibers can be added with springs at one end as necessary to maintain a minimum preload on the fixed fibers during cooldown and through any acceleration loads that are expected. These can be made long enough that they don't contribute significantly to the parasitic heat load. It would be more efficient to design a system that would maintain its preload without the extra fibers, but this is quite difficult in practice.

There are two reasons for requiring a minimum resonant frequency. One is simply to have a system that can survive expected vibrational loads and which does not contribute significantly to microphonic interference with the detectors. The usually more critical one is to minimize vibrational heat input. It is straightforward to show that the total heat input to a damped harmonic oscillator of mass *m*, resonant frequency f_0, and damping factor γ suspended from a surface driven by an acceleration g(f) is

$$P = \int_0^\infty 96\, m\, g^2(x) \frac{c/(2\pi)}{\left(1-x^2\right)^2 + c^2x^2} dx\,, \qquad (2)$$

where $x = f/f_0$, g is the r.m.s. acceleration in units of 9.8 ms^{-2} and $c = 2\pi\gamma/(f_0 m)$ $(c \approx Q)$. If the acceleration spectral density $g^2(f)$ is approximately constant over the width of the resonance (usually the case, since the *Q* of a cold Kevlar suspension is > 100), we can take g^2 outside the integral and get

$$P = \frac{96\, m\, g^2(f_0)}{2\pi} \int_0^\infty \frac{c}{\left(1-x^2\right)^2 + c^2x^2} dx\,. \qquad (3)$$

The integral is now independent of *c* and is equal to $\pi/2$ for all $c > 0$. This means there is a universal constant that determines the heat dissipation as $P = K\, m\, g^2(f_0)$, where $K = 24$ watts/kg/g^2/Hz, independent of the resonant frequency and *Q* of the system.

While one can come up with many tricky schemes that should reduce vibrational coupling, the only practical one for most complex systems is to keep the critical parts sensitive only to high frequencies. If the suspension has a higher resonant frequency than any its major supporting structures, it can be effectively isolated from external vibrations. A sample analysis for a cryostat and demagnetization refrigerator designed to stay at 50 mK through a sounding rocket launch with 17 g_{rms} vibration can be found in Cui et al (1994)[12].

Besides the obvious problem with large amounts of vibrational heating shortening hold times, there is a more subtle and potentially more serious problem with the same source. Low level vibrations, such as from mechanical coolers associated with the cryostat, can introduce small amounts of heat to the suspended cold plate. If these were perfectly constant, the temperature regulation system would simply have to run the salt pill slightly colder to keep the detectors at the desired temperature. However, beats and random modulations in the vibration level will cause time variations in this temperature increase. If these are outside the bandpass of the temperature control loop, then they will not be removed and will produce gain fluctuations in the detectors. These can seriously degrade the performance even at the microkelvin level, particularly for high resolution X-ray microcalorimeters.

Besides keeping the resonant frequency high enough that the cold stage can be effectively isolated, there is one more scheme for reducing the effects of this heat input. Although the total power dissipation depends only on the vibration level and suspended mass, we have some control over where it goes. The dissipation is independent of the damping, but it goes entirely into whatever is providing the damping. In the case of a normal Kevlar suspension, this is almost surely dominated by the Kevlar itself, and we'd expect about half the heat to go to the cold side and half to the hot. But the Q is very high, and a modest amount of added damping would soak up almost all the heat input if it could be arranged so that the dissipating part was thermally connected only to the hot side.

One possibility for accomplishing this is to mount a few grams of cobalt-samarium magnets to the cold stage. A multipole arrangement of four or eight disks each a few mm in diameter mounted close packed in a planar array will produce a magnetic field that drops off very rapidly at a distance, but is quite effective in inducing currents in a small copper plate mounted 1 mm away and attached to the hot side thermal bath. The RRR can be made high enough to be limited by the skin depth at the mechanical resonant frequency for a plate ~2 mm thick, and this provides enough magnetic damping to reduce the Q to a few. Since 99% of the damping is then in the eddy current heating of the copper instead of in the Kevlar supports, heat input to the cold stage should be reduced by a large factor.

Design and optimization of salt pills using hydrated salts: continuous ADRs

Continuous ADRs operate by repeatedly cascading heat from a "continuous stage" through each adjacent stage, to a heat sink. Since the cooling power is inversely proportional to the cycle period, obtaining high heat transfer rates between stages is a driver for system design. More specifically, the transfer between the coldest stages, at or slightly below the operating temperature of the ADR, demands high thermal conductance for both the heat switch and the salt pill. The latter, at low temperature (<100 mK), requires a much more substantial thermal bus than is needed for single-shot ADRs, as much as 25-30% of the salt pill volume.

The large volume fraction of the thermal bus suggests a need for automated machining instead of manual assembly from wires. It also suggests that the bus should be machined from common metals such as copper, instead of precious metals such as gold, and thus may have an impact on the choice of refrigerant. FAA is highly corrosive to copper, and previous attempts to use gold-coated copper wires has not been satisfactory – any imperfection in the coating leads to the complete dissolution of the underlying copper. CPA, on the other hand, is not corrosive, and although it may not always be the best choice from a thermodynamic perspective, the practical advantage of moderating fabrication and materials cost is compelling.

For the salt pills used in CADRs at NASA/GSFC[13], the thermal buses were machined using wire electric discharge machining (EDM) from cylindrical blanks of high-purity (>99.99%) copper. A subsequent brazing step (in vacuum) ensured very high RRR (>1000) in the finished pill. The bus was designed to meet the complementary requirements of high surface area (to increase thermal boundary conductance) and high A/L along the length of the salt pill (to increase axial conductance). As shown in Figure 5, the bus is formed as a grid of copper conductors by two series of cuts along the axis. The conductors were rectangular in cross section, with dimensions Δw_x by Δw_y, with center-to-center spacings of Δx and Δy.

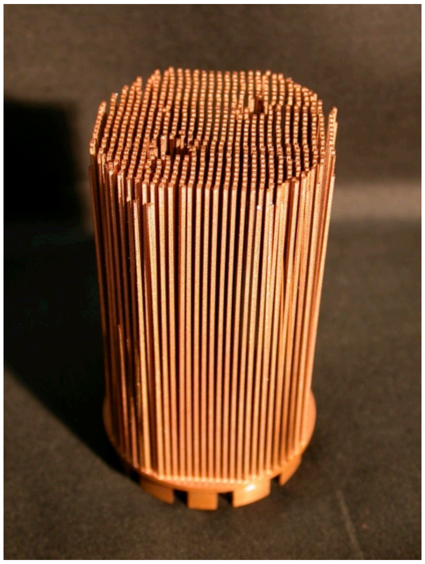


Figure 5. Wire EDM machined thermal bus for low temperature salt pills. The axial holes will later be used in the salt growing process.

For a salt pill of radius *r* and length *L*, the number (*N*) of conductors, their surface area (*A*) and their volume fraction (*f*) are

$$N = \frac{\pi r^2}{\Delta x \Delta y} \tag{4}$$

$$A = 2N\left(\Delta w_x + \Delta w_y\right)L \tag{5}$$

$$f = \frac{N\Delta w_x \Delta w_y}{\pi r^2} = \frac{\Delta w_x \Delta w_y}{\Delta x \Delta y} \tag{6}$$

Characterizing the thermal conductance between the bus and salt is not straightforward, since heat absorption (or generation) is distributed along the length of the salt pill. A simplifying assumption is that the thermal bus and salt are isothermal, with temperatures T_{bus} and T_{salt}, and that the difference between the two is the same as if all heat entering the salt pill ($\dot{Q}$) travels half the length. The corresponding boundary (K_B) and thermal bus (K_{bus}) conductances are

$$K_B = \frac{A T_{bus}^3}{R_B} \tag{7}$$

$$K_{bus} = \frac{2k(T_{bus}) N \Delta w_x \Delta w_y}{L} \tag{8}$$

and the gradient between the bus and salt is then

$$T_{bus} - T_{salt} = \dot{Q} / \left(\frac{1}{K_B} + \frac{1}{K_{bus}} \right) \tag{9}$$

where R_B is the coefficient of the thermal boundary resistance and k is the thermal conductivity of the bus material at T_{bus}. For copper, k~(RRR/60)•T_{bus} in W/cm•K. While thermal boundary resistance has been measured for some combinations of salt and bus material that were common in the early days of ADRs – for example, for CPA in contact with varnished copper wires[14], R_B~30 K^4•cm^2/W – we expect that alums in contact with bare metals will have boundary resistances comparable to the value calculated by Little [15] for CPA and Au: R_B~17 K^4•cm^2/W.

The mass of salt (assuming 100% fill fraction in the available volume) is $m_{salt} = (1-f)\rho\pi r^2 L$, where $\rho$is the bulk density of the salt. The cooling capacity can be computed from the standard entropy function, modified to include nearest neighbor interactions [16]. The entropy points that define the capacity are zero field at T_{salt}, and peak field, B_{peak}, at the temperature where the stage is recycled, $T_{recycle}$:

$$\Delta Q = m_{salt} T_{salt} \left(S(0, T_{salt}) - S(B_{peak}, T_{recycle}) \right) \tag{10}$$

To a large extent, only the zero-field entropy term, with its dependence on the salt temperature, will impact the optimization of the thermal bus. The entropy at the peak field is simply a reference value for calculating the gross cooling capacity.

Optimization of the thermal bus involves selecting values for Δx, Δy, Δw_x, Δw_y that maximize ΔQ. Other parameters (r, L, $\dot{Q}$, T_{bus}, B_{peak}, $T_{recycle}$, and the RRR of the copper bus) are regarded as fixed, but may in fact be adjustable to achieve optimization at a higher level of assembly, or to build in margin on performance. For instance, the aspect ratio ($L/2r$) is important both for its affect on axial conductance and in setting the size scale (and mass) of the magnet and any magnetic shielding. If one considers only the latter [17], stage mass is minimized for an aspect ratio of approximately 2.75. However, the minimum is broad and shallow, and for salt pills where axial conduction is a limiting factor, smaller aspect ratios may be chosen to improve thermodynamic performance.

It should also be noted that while making the thermal bus more finely divided (having a larger number of smaller conductors) will increase the boundary conductance and improve cooling capacity, there is a practical limit based on machinability. The final dimensions may need to be a compromise between performance and machinability, or, more likely, the cost and time of fabrication.

To illustrate the process, we use the example of salt pills developed for a 4-stage CADR operating at 50 mK with more than 5 microwatts of cooling power[13]. The coldest stages were designed for a cooling capacity on the order of about 50 mJ at 50 mK, and maximum heat fluxes of 40 μW. Table 1 summarizes the design parameters. The peak field and recycle temperature for this stage are 0.6 T at 0.3 K.

Table 1. Optimized parameters for small CPA salt pills used in a 4-stage CADR

Parameter	Pre-determined Value	Optimized Value
Maximum heat flux (μW)	40	
Temperature, T_{bus} (K)	0.05	
Radius, r (cm)	2.0	
Length, L (cm)	6.4	
Thermal bus material	Copper, RRR>300	
Conductor dimensions (cm)		0.051 x 0.051
Center-to-center spacing		0.13 x 0.13
Number of conductors		740
Surface area, A (cm^2)		1000
Volume of salt pill (cm^3)	80	
Volume of thermal bus (cm^3)		12.8
Salt fraction		84%
Salt mass (g of CPA)		120
Thermal conductance, K_{bus} (W/K)		0.076
Boundary conductance, K_B (W/K)		0.0074
T_{salt} with 40 μW heat flux (K)		0.0441
Cooling capacity at T_{salt} (mJ)		57.6

The "optimal" dimensions for the thermal bus conductors tend toward the smallest possible size with a 3 times larger center-to-center spacing – corresponding to a salt fill fraction of 88%. In the above example, the dimensions chosen were at the limit of the EDM machine's ability to produce consistent conductor widths, in part due to the distortion of the thin conductors during the cutting process as a result of internal stresses in the bulk copper. They also reflect uncertainty in the magnitude of the thermal boundary resistance, and hence tended toward larger surface area at the expense of salt mass. Nevertheless, the cooling capacity obtained is approximately 80% of the maximum value possible in the limit of infinitesimally small conductors.

Hermetic Enclosure and Salt Growth

For salt pills using this design, a hermetic enclosure can be formed by a brazing step that attaches a copper cap to the free end of the copper conductors, stainless steel collars to the openings where salt solution can be introduced, and stainless steel rings at each end of the assembly. The brazing process also serves to anneal the copper bus. An outer stainless steel sleeve is then welded to the rings to form a rigid, hermetic structure, as in Figure 6.

The salt is grown in situ using the two openings, through which saturated solution can be introduced and depleted solution can be withdrawn. Details of this process have been described previously[2, 6, 18]. Once the salt growing is complete, small caps are welded into the openings and the salt pill is gold coated.

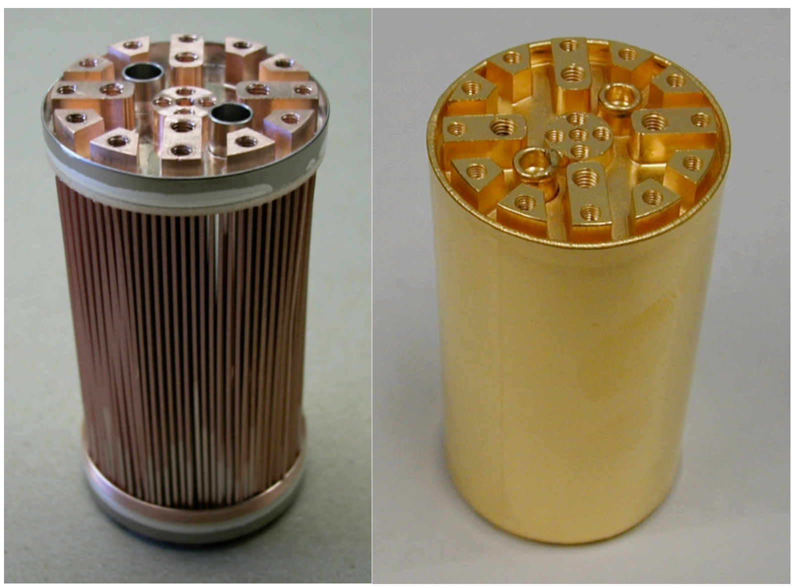


Figure 6. Formation of the hermetic enclosure for salt pills using EDM machined thermal buses.

Design and optimization of salt pills using single- and poly-crystals

ADR stages operating at temperatures above about 0.3 K can take advantage of higher density materials that are, in many ways, more robust and stable than hydrated paramagnetic salts. The most common are Gd-based compounds, manufactured as single-crystals (grown from a melt [19]) or as poly-crystals (synthesized from powder [20]). These have significantly higher magnetic ion densities, and therefore support much higher cooling powers and heat fluxes.

With a few exceptions, these materials are produced in bulk using processes and temperatures that preclude synthesizing the crystals directly onto a thermal bus. Any features needed, for example to make connections to the bus, must be ground or machined into the finished crystals. Thermal buses for these materials therefore tend to be as simple as possible. The two most common strategies are the use of compression/bonded contacts to flat areas on the crystal, and the use of hermetic enclosures in which exchange gas provides thermal contact.

Compression/Bonded Contacts

For many applications, such as those in which only small to moderate heat flows are required, mechanical and thermal contact to a crystalline salt can be made using compression or bonded joints. In these cases, thermal conduction within the salt and across the interface yield sufficiently small gradients between the external interface and the bulk salt that heat absorption efficiency remains high.

Compression joints are inherently more stable over time and with thermal cycling, but with typical components – soft copper – it is difficult to achieve large compressive force, and therefore joint conductance tends to be very limited. In applications that do not require heat transfer between stages, such as the single-magnet 2-stage ADR commercially available from High Precision Devices, Inc.,[21] peak heat loads are in the microwatt range, and the small joint conductance is more than adequate. For ADRs where the upper stage is used to pre-cool a lower stage, heat flows in the milliwatt range are needed, and bonded joints are a better choice.

The challenge for bonded joints is long-term stability. The stresses induced from differences in thermal contraction can cause microcracking and eventual joint failure. Proper selection of mating material and bonding agent is therefore crucial.

The interface material should be chosen to match the overall thermal contraction rate as closely as possible. As an example, for GGG, integrated thermal contraction to low temperature depends on the crystal orientation[22]. It is largest along the <100> direction, at 0.10%; in the <110> and <111> directions the contraction is 0.064% and 0.069%. While small compared to typical cryogenic materials (copper, aluminum, etc.), these values are comparable to that of tungsten (0.089%)[23]. High purity tungsten also has high conductivity, making it a good choice for an interface to a GGG salt pill. Figure 7 shows two GGG salt pills used built for use as the upper stage of 2-stage ADRs.

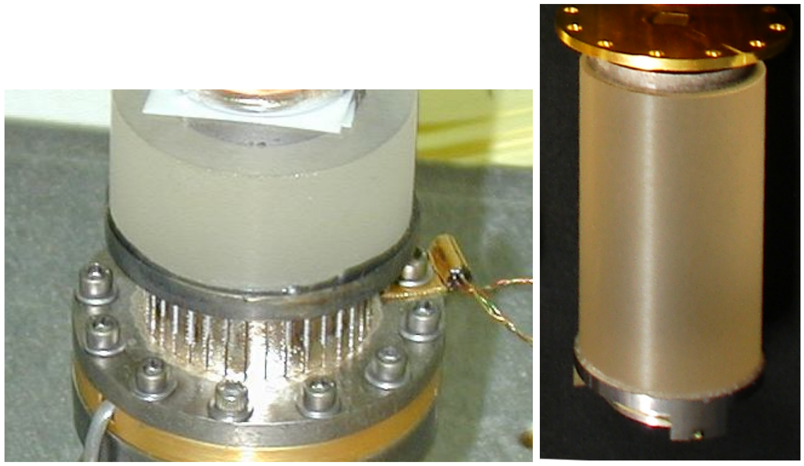


Figure 7. GGG crystals bonded to a tungsten thermal interfaces.

The choice of bonding material is also critical, but less so when the thermal contraction of the salt pill and interface are well-matched. A range of epoxies have be used with excellent results, including Stycast 2850FT and Scotchweld 2219 (Figure 7). For thin, well-controlled bond lines, the thermal conductivity of the epoxy is generally not a limiting factor in the selection.

Hermetic Enclosures

When high heat transfer rates are required, or when using materials with high thermal boundary resistance or poor thermal conductivity, thermodynamic performance can be significantly improved by encapsulating the salt in a thermally conductive, hermetic enclosure filled with helium exchange gas.

Such structures have been used in both continuous ADRs[5] and in the upper stages of the 3-stage ADR developed for the Soft X-Ray Spectrometer (SXS) instrument on Astro-H[24]. In these systems, heat flows between stages are as large as 5-10 mW in the 1-1.5 K range. Moreover, the material chosen for the SXS stages was gadolinium lithium fluoride (GLF), based on its 30% higher entropy capacity per unit volume compared to GGG[19]. The material was only available in polycrystalline form, with approximately an order of magnitude lower thermal conductivity than single-crystal GLF[25].

The basic structure is a thin-walled tube section connected to end fittings. Generally there is no need to make thermal contact at both ends, so one end extends into a thermal interface, and the other contains a cap and filling port. The salt pills developed for SXS are shown in Figure 8. The enclosure is a two-part brazement, consisting of a high purity copper canister (slotted for eddy current reduction; see below) with integral bolting flange, and a stainless steel sleeve. The brazing of the two parts acts also to anneal the copper. After the GLF wafers are loaded into the salt pill, an end cap is e-beam welded into position. The end cap contains a CuNi tube into which a soft copper tube can be soldered. The copper tube connects to a filling system, through which helium gas can be admitted. Afterward, the tube is crimped and solder sealed.

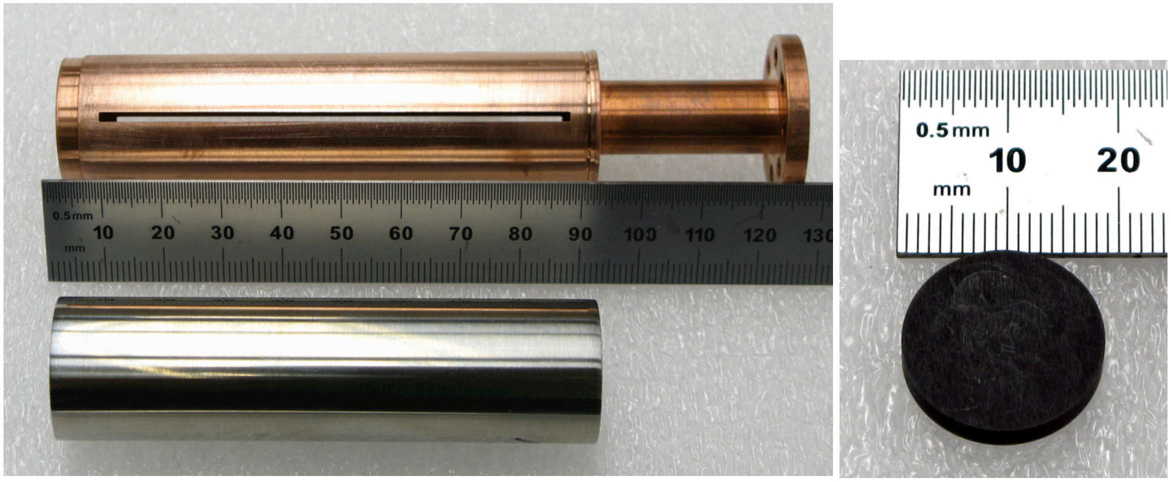


Figure 8. Hermetic enclosure for the SXS salt pill, using wafers of GLF (right).

Since the operating range of stages using GGG or GLF often extends below 1 K, but rarely below about 0.5 K, ^{3}He gas can be used to provide effective heat exchange. The gas charge at room temperature is set by the goal of keeping the mean free path small enough (≤1% of the gap between the refrigerant and the container) for the gas to be in the viscous limit. However, below the temperature where the gas condenses into a thick film, the mean free path will be dictated by the saturated vapor pressure. For a 1 atmosphere initial charge, the condensation point is ~0.74 K, where the pressure is ~2 torr, corresponding to a mean free path of less than 0.1 μm. The mean free path is essentially constant at higher temperature, but increases at lower temperature due to the rapidly decreasing vapor pressure. Since gaps smaller than 20-50 microns are not practical, a 0.5-1 atmosphere charge satisfies the criteria for mean free path down to the point where it becomes intrinsically limited by condensation effects.

Performance of salt pill enclosures can be difficult to measure since there is generally no direct way to probe the gradient between the salt and interface. However, an estimate can be made by imposing a discrete change in heat load under constant field conditions. The salt is effectively isothermal, and the change in interface temperature reflects the gradient to the salt. Such measurements have been made for the SXS salt pills (which contain a 8.1 cm stack of 1.9 cm diameter GLF wafers, and use a copper wall thickness of 0.5 mm), giving end-to-end conductances of ~250 mW/K at 1.3 K. By itself, the copper canister would be expected to have a conductance in excess of 1 W/K.

Eddy Current Dissipation in Thermal Buses

While it is almost always necessary to have some electrically conductive material in the magnetic field region of a stage, care must be exercised to prevent eddy currents from degrading thermal performance. This is especially problematic in higher-temperature stages where the thermal bus is concentrated into larger scale structures than the finely divided buses used with hydrated salts.

Eddy current dissipation in a cylindrical tube of radius *r*, thickness Δ*r*, and length *L* in the presence of a changing magnetic field $\dot{B}$ is:

$$\dot{Q}_{tube} = \frac{\pi L \dot{B}^2}{2\rho} r^3 \Delta r \tag{11}$$

where ρ is the resistivity of the material at low temperature. For solid rods of radius *r*, the last expression can be converted to differential form and integrated over the radius to yield

$$\dot{Q}_{rod} = \frac{\pi L \dot{B}^2}{8\rho} r^4 \tag{12}$$

To give some idea of the magnitude of the eddy current dissipation relative to the cooling capacity of a stage, consider a containment tube of thickness 1 mm and *RRR*=300 (representative of annealed OFHC copper) surrounding a GLF pill of radius 1 cm. For representative cycle parameters (4 T peak field, and a heat sink at 4.2 K), the entropy capacity at 1 K is 0.33 J/K/cm.

If the salt pill is demagnetized from 4 T at 4.2 K to ~1 T at 1 K in a 5 minute period, eddy current dissipation per unit length will be 0.030 mW/cm. Since this heat is absorbed as the temperature is steadily decreased, the entropy generation increases as the salt cools, as the inverse of temperature. The entropy change, integrated over the 5-minute demagnetization, is 0.0039 J/K/cm, or about 1.2% of salt's capacity.

Eddy Current Mitigation

There are many effective strategies for reducing eddy current heating. In many instances, the removal of small amounts of material – to interrupt current loops – will reduce or eliminate eddy currents without significantly affecting the thermal conductance of the bus. For example, a lengthwise cut using wire EDM through a solid rod will reduce the dissipation by more than a factor of 2.

A similar cut (see Figure 8) in a cylindrical tube can reduce dissipation by 2-3 orders of magnitude, since currents are then limited to circulating only within the c-shaped cross section of the shell. As a result, the relevant flux is that contained within the conducting material, not the flux within the bore of the tube, and the longer, narrower flow path for currents yields a significantly larger resistance. However, bridging currents at the ends of the slot can be a problem, and more detailed modeling of the geometry may be needed to assess the resulting eddy current losses. In any event, though, the tube's role as a hermetic container is compromised. For SXS, the combination of a slotted copper canister and brazed stainless steel sleeve achieved both low eddy current losses and hermetic containment of the exchange gas.

SUMMARY

Over the last 3 decades, ADR designs and capabilities have undergone a dramatic expansion. Multi-stage ADRs are now common, with higher cooling capacities and wider operating temperature than is possible with single-stage systems. Salt pill designs have evolved also, to accommodate higher heat flows, and to incorporate newer materials that can be used in warmer stages. For low temperature ADR stages, where the use of hydrated salts is required, the design still centers on packaging the salt in a hermetic container (to prevent dehydration), and on the creation of a thermal bus that conducts heat to/from the salt. Its optimization involves trading off salt volume for higher conductance to yield the highest cooling capacity at the operating point. Designs appear to be converging on all metal-sealed units, with either hand assembled (gold) wire buses, or buses cut using automated machining techniques such as wire EDM.

For higher temperature materials, which as bulk crystals generally do not require any special packaging, mechanical and thermal contact to the salt can be achieved in a variety of ways. The simplest is to use a compression joint to a metallic interface, and this is satisfactory for stages that require only modest heat flows. For ADRs where high heat flows are required, or where the materials used have poor thermal conductivity, dedicated thermal buses are thermodynamically advantageous. One approach that has been used extensively in multi-stage ADRs is to encapsulate the material in a conductive can filled with exchange gas. Using ^{3}He provides excellent heat exchange down to 0.3 K, which is about the physical limit for materials such as GGG and GLF.

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