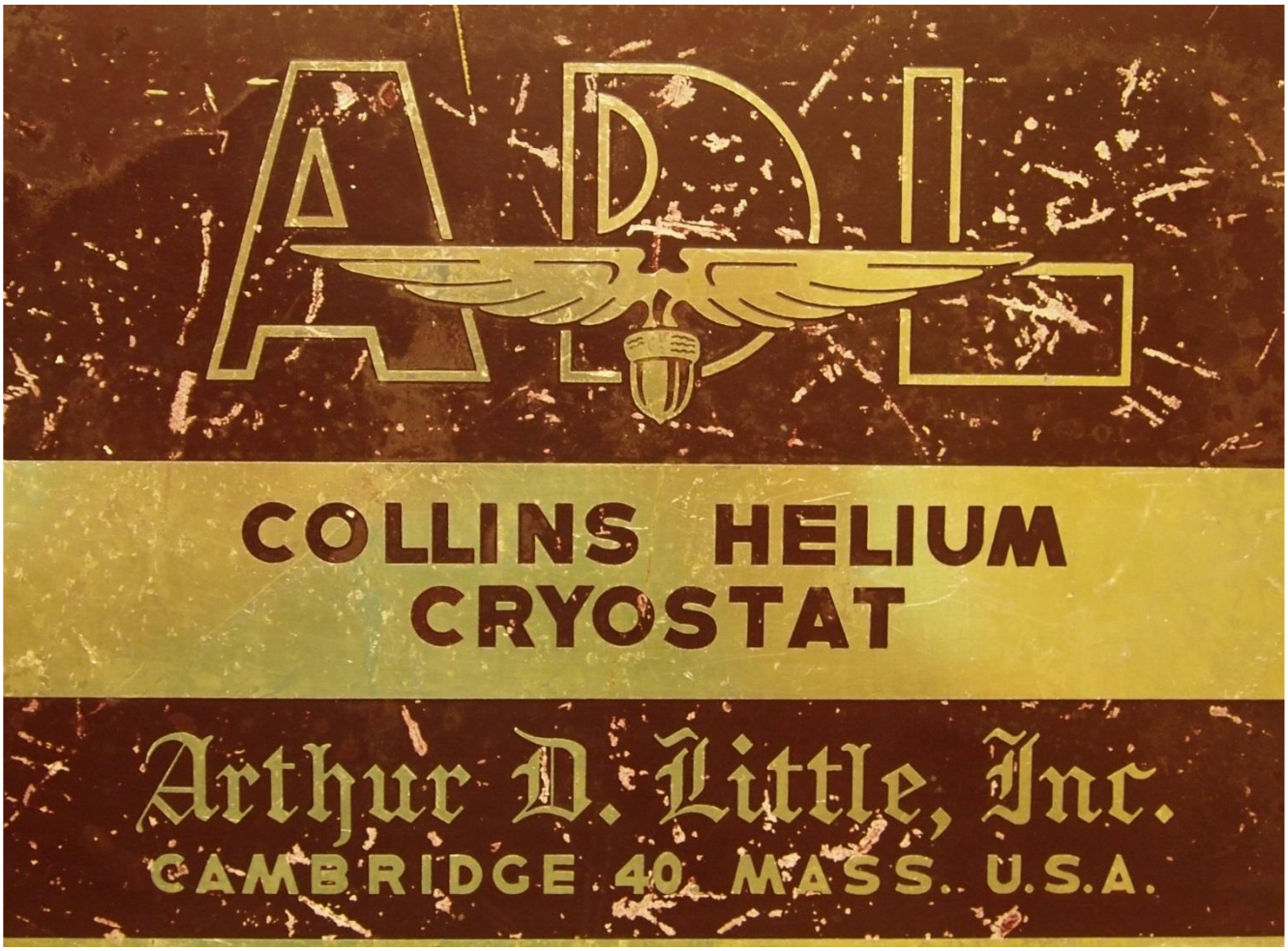

History of the Low Temperature Laboratory

Iowa State University – D. K. Finnemore -- 2017

The idea to ask Doug Finnemore to write the history of the Low Temperature Cryogenic Laboratory came during our morning coffee with Sergey Bud'ko. With forthcoming retirement of Marc McGinn, all current personnel of the lab is notably disconnected from the history of (rephrasing Isaak Newton) "Giants, we stand on shoulders of". Doug covered perfectly the story of the lab approximately till 1997, when latest Model 1410 Process Systems Intl. liquefier was installed and about the time when Jerry Ostenson retired and Sergey Bud'ko took the lead. At about the same time, both in Ames and globally, the era of low temperature measurements as an artisanal endeavor of selected few, gave place to an all-inclusive epoch of measurements using highly automated standard instruments leading to a notable increase of cryogen demand. The follow up developments in the Low Temperature Cryogenic Laboratory included change of the liquefier compressor in 2003 and its computer control in 2016, complete replacement of the PVC piping of the He recovery line with copper tubing (finished at about 2010), upgrade to Helium recovery compressors, purchased in 2003 and 2011, upgrade of the liquefier electronics in 2016, installation of recovery lines in Sensitive Instruments Facility in 2017, installation of high pressure liquid nitrogen tank in 2016 and construction of high purity nitrogen gas line to service DNP NMR experiments of Marek Pruski laboratory.

Doug Finnemore was modest enough not to mention much his own work, but one quotation I need to add for the next generation to remember. Comparing cooling with liquid nitrogen and liquid helium, Doug compared this as cheap beer versus expensive scotch.

Makariy Tanatar

Ames,

January 30, 2018

History of the Low Temperature Laboratory

Iowa State University – D. K. Finnemore

2017

Introduction

At Iowa State University, most of the early research at liquid helium temperatures was carried out on the rare earth and actinide elements of the periodic table. During World War II, Frank Spedding and Harley Wilhelm had developed commercial scale processes to purify uranium and had transferred these methods to industry. In the course of doing this work, they also learned how to produce high purity samples of both uranium and thorium as well as developing methods to separate the rare earth elements using ion exchange columns. An important part of this work was the development of the analytical techniques to detect impurities and verify the quality of the materials. Samples of these high quality rare earth materials were made available to researchers around the world giving Iowa State an international reputation as a center for rare earth research. At a physics colloquium in the 1970s, Wally Koehler from Oak Ridge once observed, somewhat tongue in cheek, "Rare earth research may not be mandatory in Ames, but it certainly is rampant." If you worked with rare earths in Ames, you had a competitive advantage because you had the best materials in the world.

With the formation of the Ames Laboratory in February of 1947, there was an increased effort in the study of the actinides and rare earth materials and an overall expansion of work to solve problems for programs growing out of the Atomic Energy Act of 1946. Chemistry, metallurgy, condensed matter physics and engineering all benefited from these expansions. Several buildings were constructed to house the work of the Ames Laboratory including the Office and Laboratory building (the Link) between Chemistry and Physics Hall in 1947, Wilhelm Hall in 1947, Spedding Hall in 1949 and Metals Development Building in 1959. There was a strong interest in establishing a major materials laboratory with a strong emphasis on analytical chemistry to determine the impurities and quality of the materials being prepared as well as the study of physical properties over a wide temperature range. At about the same time, the transistor was invented in December of 1947 opening the field of semiconductors. In addition, at the National Bureau of Standards in Boulder, there had been a major effort to study liquid hydrogen, deuterium and tritium as part of the nuclear and hydrogen bomb program so there was a push to study the properties of materials at low temperatures. Possibly most important, Sam Collins at MIT was developing a helium liquefier that would soon make liquid helium available around the world. All of this work stimulated new research at low temperatures.

Early research

In an early research project at helium temperatures, Frank Spedding sent samples of lanthanum, cerium, praseodymium and neodymium to Francis Simon at Oxford University, a leading European university in cryogenics, where they measured the specific heat from 2K to 180K.[1] In Simon's lab, they liquefied helium in the same dewar that they used for the specific

heat measurement. They cooled high pressure helium gas with liquid hydrogen and then expanded the high pressure helium gas to make small amounts of liquid, routinely reaching 1.5K with this method. For lanthanum, they found a superconducting transition at 4.37K indicating that the sample was primarily in the hexagonal phase. For the other rare earths, they found anomalies at both the crystallographic and magnetic transitions. In Ames at about the same time, Sam Legvold's student, Nancy James, measured the electrical resistivity of the same four elements from 2.2K to room temperature.[2] In these experiments, the Legvold group used a Collins helium cryostat located in the northwest corner room of Physics Hall to generate the liquid helium. These Collins cryostats had a wide mouth dewar about 250 mm across with the heat exchangers and expansion engines located on one side so that there was a space wide enough to accept a resistivity or other cryostat inserted along the other side of the dewar. Small pieces if Styrofoam were placed in the helium space so that you could look into the dewar from the top and see the Styrofoam floating on the liquid helium. Hence, with a Collins cryostat, experiments could be done either directly in the liquefier cryostat or helium could be transferred out to another dewar where the experiment would be done. Somewhat later in Elliott's study[3,4] of magnetic susceptibility from 20K to room temperature of gadolinium in 1953 and of neodymium in 1954, a temperature controlled gas stream was used to provide isothermal conditions in a one Tesla iron core magnet. Here, helium clearly was transferred out of the Collins helium cryostat.

During this same time period, Griffel , Skochdopole and Spedding[5] in chemistry were publishing the specific heat of Gd and Th down to 15K in 1953. Maurice Griffel was a low temperature chemist with a Ph. D. from the University of Chicago in 1949 and an assistant

professor in the Chemistry Department at the time. Skochdopole was his student. For this work, there was a second Collins liquefier in 227 Spedding Hall.[6] Presumably, at some later time, this second Collins helium cryostat was moved to Room 0012 Physics Hall and was converted to a hydrogen liquefier.

Over the succeeding fifteen years, the Ames and Oak Ridge groups were to team up for an in-depth study of magnetism in the rare earth metals. Spedding's group prepared all of the rare earth metals in very pure form, and the Legvold group learned to prepare centimeter size single crystals suitable for neutron scattering. The Legvold group used a strain anneal method in which a button of high purity rare earth metal would be put in a mechanical vice to introduce a large amount of strain energy. Then the strained button would be annealed for weeks or months in high vacuum to relieve the strain and grow large single crystals. The Legvold group first mapped out the basic thermodynamic and transport properties of these single crystals as a function of temperature and magnetic field to locate the phase transitions and fit the results to current theory. Then the crystals were shipped to Oak Ridge National Laboratory for a neutron scattering study of the detailed magnetic spin structures.

Personnel

Sam Legvold supervised the liquid helium service in the early going, and the equipment was located in the northwest corner room of Physics Hall, the space now occupied by the main Department of Physics and Astronomy office called Room 0012. Sam took a B.S. degree from Luther College in 1935 and a PhD from Iowa State in 1946 studying under J. V. Atanasoff of digital computer fame. He was a contract employee with Naval Ordinance during the middle of the war and then returned to Iowa State. Sam hired Ralph Houchin as a Senior Technician to run the Collins Liquid Helium Cryostat and Tom Naig as a machinist shared between the

Institute of Atomic Reseach and a University Machine shop supervised by Ike Coleman in the space in Physics Hall where Rooms 0091 and 0092 are located.

Fig. 1 Bill Sylvester, Dave Nelson, Mike Sandholm, xx,yy, Sam Legvold, Roger Edwards, zz, Paul Ness, aa, Virgil Banowitz, cc, Don Boys in 1963.

The addition of Clayton Swenson to the physics faculty in 1955 provided a substantial influx of low temperature expertise. He received a D. Phil. in 1949 with Francis Simon at Oxford, doing a dissertation studying the solid-liquid phase boundary in helium near absolute

zero.[7] During his time at Oxford University, Clayton also published several papers on low temperature methods in *Reviews of Scientific Instruments*. He then spent three years as an instructor at Harvard helping them set up a liquid helium temperature facility using a Collins helium liquefier from Arthur D. Little.[8] He then took a job with Sam Collins at MIT for three more years on a federal post-doctoral appointment. Bille Carlson, a nuclear theorist at Iowa State, had been a friend of Clayton at both Harvard and at Oxford and partly through Bille's influence, Clayton moved to Iowa State in 1955.

Fig. 2 Sam Legvold is discussing an experiment with a student in the left picture, and Clayton Swenson is working on a cryostat in the right picture.

In the succeeding years, Paul Ness was hired November 7, 1960 and Mike Sandholm was hired September 18, 1961 to replace Ralph Houchin. Doug Finnemore was hired on 8 June 1962 and Jerry Ostenson started on 1 July 1962. Marc McGinn was hired to replace Mike Sandholm in 2001 and Bob Howard was hired in February of 2015 to replace Bill Wing who replaced Paul Ness.

Operation of the Low Temperature Laboratory in Physics Hall before 1967

The original low temperature laboratory was located in the northwest corner of Physics Hall in Room 0012 and was organized as shown by the sketch in Fig. 3.

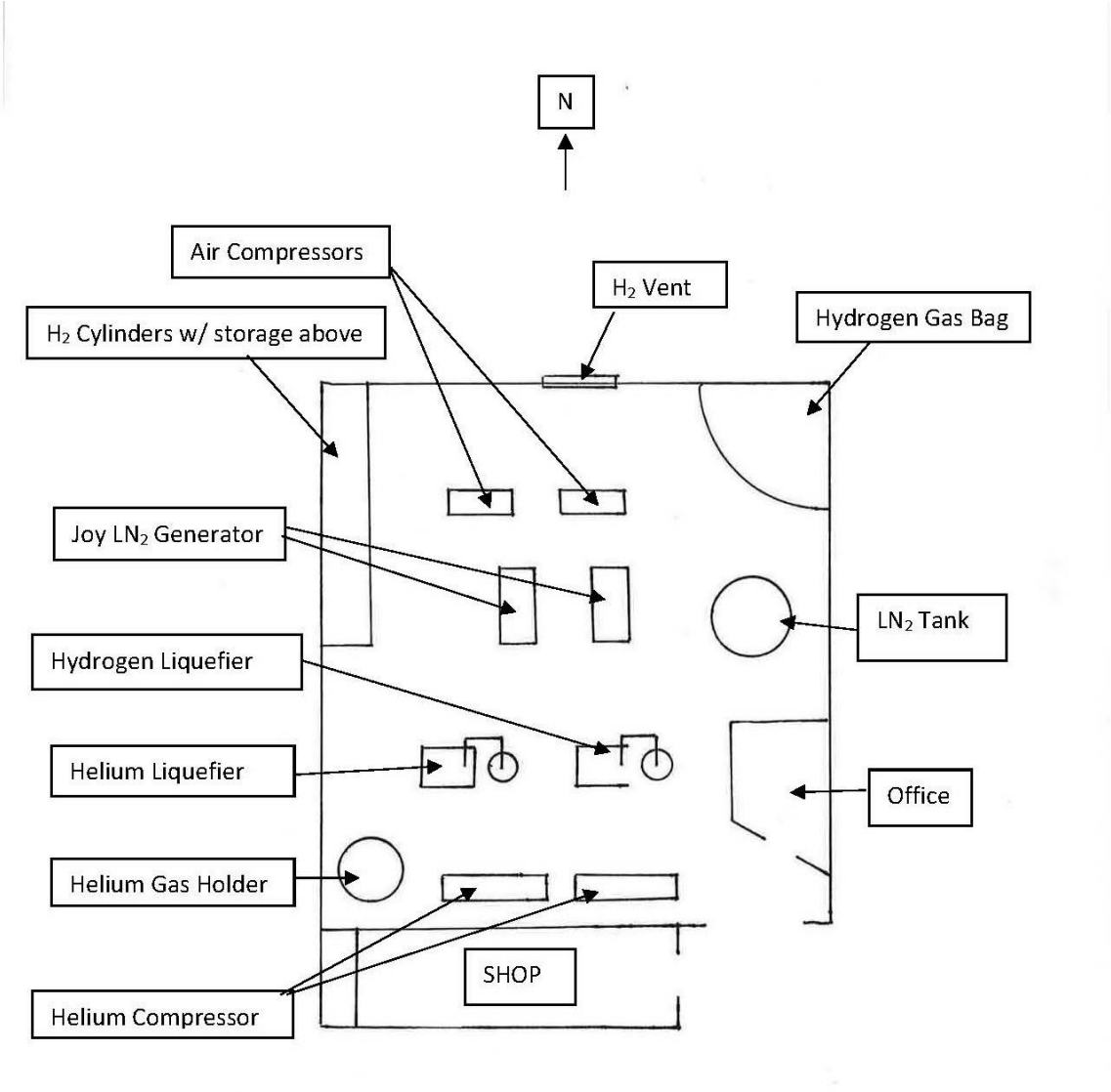

Fig. 3 Sketch of the Low Temperature Laboratory in Physics Hall in about 1964.

As you walk in the south door of Room 0012, the helium compressors were just to the left and a small office was located straight ahead. The Collins helium liquefier and the Collins liquefier

that had been converted to liquefy hydrogen were just north of the helium compressors. A helium gas holder used for short term storage of helium gas was located in the southwest corner of the room. It was a large cylindrical drum with a neoprene bladder or bag that rose as helium flowed into the bag. It was equipped with a line and pulley that would control a switch that would open the bag to the bank of helium cylinders from which helium could flow if the gas supply was low. Another switch cut off the flow when the gas holder became full. There were two liquid nitrogen generators that had been purchased from the Joy Manufacturing Company located north of the helium and hydrogen liquefiers. They delivered liquid nitrogen into a 1000 liter storage tank along the east wall. Two air compressors that fed the nitrogen liquefiers were just north of the nitrogen liquefiers. Along the north end of the west wall was a row of hydrogen cylinders to supply hydrogen gas to the hydrogen liquefier when needed. There was a vent along the north wall and a hydrogen gas bag in the northeast corner to provide a temporary storage place for excess hydrogen gas.

If you were to look out the north window of the low temperature laboratory in those days, you might see three Cadillacs with whopping big tail fins parked in the spaces along the shed that housed the Ames Lab business computers. Two of these Cadillacs would be new and belonged to Frank Spedding and Rollie Good, a theoretical physicist. The third would be a few years older and belong to Sam Legvold. Sam was often chided about owning such a big car and he would remind us of "The law of conservation of trucks and busses," which one assumes had something to do with conservation of momentum in a collision. Sam liked to buy Cadillacs that were either four years old or had 100,000 miles on them and drive them until they died, thus providing him with a smooth ride to Deer Lake in Minnesota where he owned a cottage.

Early Educational Outreach

Sam Legvold assembled a kit of liquid nitrogen experiments that could be performed for a public audience such as grade school children or a VIESHA demonstration. He might fill a balloon with carbon dioxide gas from a fire extinguisher and dip the balloon into a wide mouth dewar of liquid nitrogen to show the volume contraction of gasses of about 700 to 1 on solidification. The solid CO_2 makes a good rattle inside the rigid balloon and provided an opportunity for a little patter about the ideal gas law and the average distance between molecules in a gas or in the solid phase. He would then set the balloon on the bench to warm up while proceeding to the next experiment. He might take an electrical circuit composed of a battery, light bulb and a length of insulated copper wire wrapped around a wood dowel to show the change in electrical resistance of copper with temperature. The resistance at room temperature was adjusted so that the light bulb glowed very dimly, but when he dipped the copper wire in a dewar of liquid nitrogen, the light bulb would glow brightly. He usually also did the flip-side experiment with a similar arrangement hooked to a semiconductor that would increase in electrical resistance with a dip in liquid nitrogen. Thermal expansion was demonstrated with a brass ball and iron ring machined so that the ball would just fit through the ring at room temperature but would not fit when the ring was at nitrogen temperature. To show the elastic-to-brittle change in ordinary substances, Sam might dip a bundle of parsley in the liquid nitrogen and pulverize the parsley in his hand. A real crowd pleaser would be to bounce a rubber ball about the size of a tennis ball on the floor a few times and then freeze the ball in liquid nitrogen. He would then throw it against the wall or floor where it would shatter into dozens of pieces. These balls were called “Pennsylvania Pinkies.”

By connecting an oxygen cylinder by a flexible hose to a test tube, he might make a little liquid oxygen by dipping the tube filled with oxygen gas into a dewar of liquid nitrogen. Typically, he would show the blue color of the liquid oxygen to the audience and pour a little liquid into the pole pieces of a small but substantial narrow gap permanent magnet where the magnetic liquid oxygen would be visibly trapped in the gap. If he really wanted to produce "oohs" and "aahs," from the audience, he might make a somewhat larger batch of oxygen so that he could soak a cigar with oxygen. He would put the cigar on the end of a heavy copper wire about a meter long and saturate the cigar with liquid oxygen. With a lighted match on the end of another 20 centimeter stick, he would light the cigar and grin with amazement as the cigar burned like a jet engine. Safety people would certainly not permit this demonstration these days.

Another demonstration involved a weight attached to a coil of lead-tin solder. If you hang a weight on the coil at room temperature, the coil will just sag letting the weight drop. If you put the coil of lead-tin solder in liquid nitrogen, then the coil is quite springy. In another magnetism demonstration, he would use a rare earth like terbium with Curie temperature of 222K to show magnetic behavior at nitrogen temperature and non-magnetic behavior at room temperature. Gadolinium has a Curie temperature of 292K so this experiment can be done with warm and cold water with a little commentary about magic followed by drinking the water.

Instrumentation in the early days

During the 1950s and 1960s, a great deal of the equipment and instrumentation used in low temperature research was designed and built in local machine and electronics shops. Commercial instruments for measurements generally were not available. For example, many postdoctorals and Ph. D. students would design and build a one of a kind cryostat to perform a specific set of measurements for their research and there were large and active machine and electronics shops to support this activity. Glass dewars were used as often as metal dewars and these dewars were readily silvered to specification in the Chemistry Glass Shop. We usually used narrow slots on opposing sides so that the researcher with a flash-light could look inside and see the liquid levels. For a public demonstration of the fountain effect, we half silvered some glass dewars and projected the gushing fountain of superfluid helium through an old-fashion slide projector onto the wall. If metal dewars were more appropriate, then they often were built in the Ames Laboratory shops to the researcher's specification.

One particular instrument used widely by most all groups was a style of rigid helium transfer tube designed by Swenson and constructed by the low temperature laboratory personnel as they had spare time for such projects. Each experimental group would have one or two of these and they were to become the standard piece of equipment built by the Low Temperature Laboratory personnel for all research groups.

Before the era of commercial flexible transfer tubes, rigid transfer tubes like the one shown in Fig. 4 were constructed by personnel in the Low Temperature Laboratory. Once the desired dimensions

of the transfer tube were known, a suitable length of thin walled Cu tubing, about 19 mm (3/4”) in outer diameter, was cut to an appropriate length, typically a length of 1500 mm. The Cu was then annealed

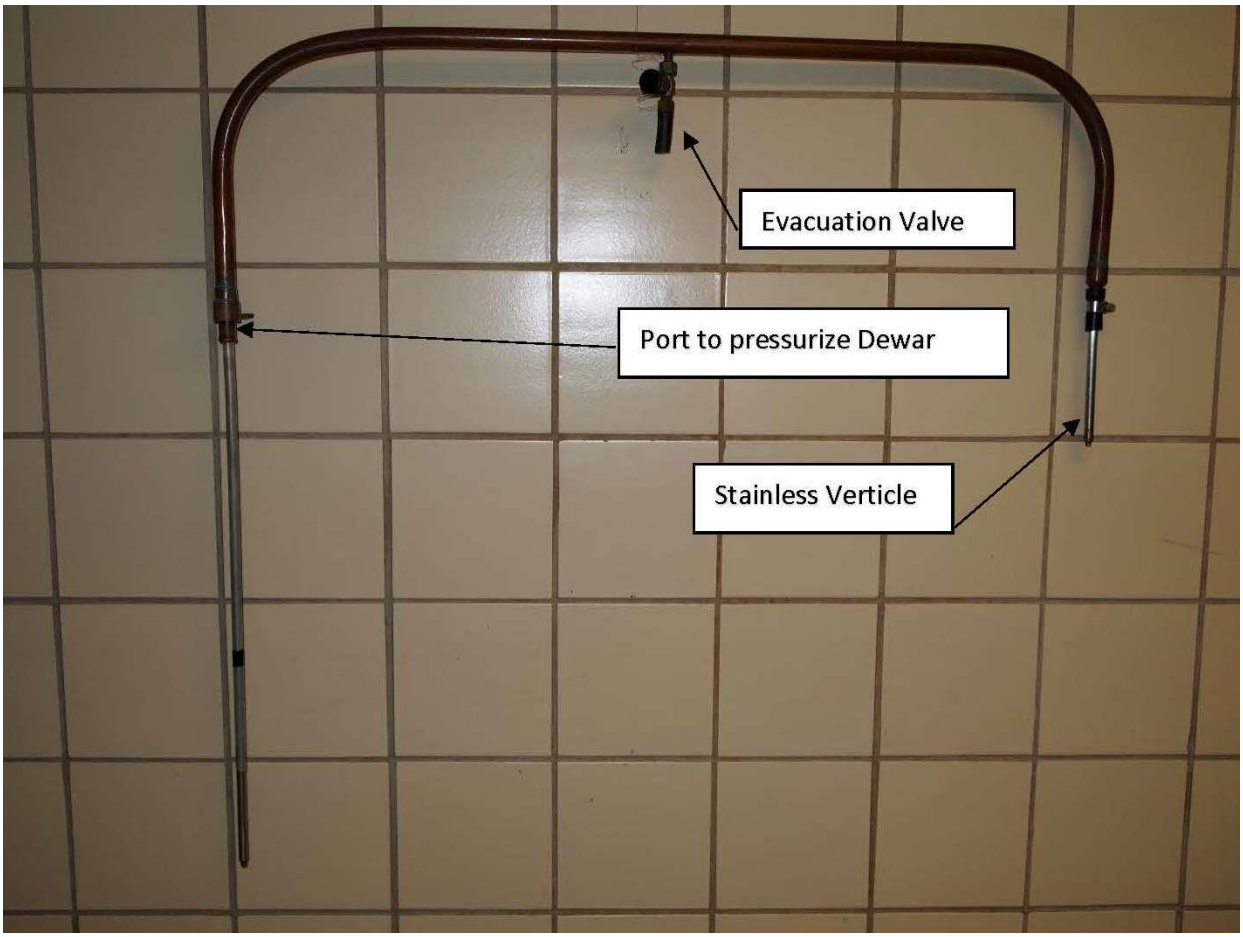

Fig. 4 Rigid helium transfer tube constructed by Paul Ness and Mike Sandholm. This example has a bayonet on the right side to be inserted into the top of the cryostat rather than going all the way to the bottom of the cryostat as was usual.

with a torch to red hot temperatures and cleaned. A rubber stopper was placed in one end of the tube, and the tube was filled with carborundum powder to keep the tube from collapsing when it was bent. After a second rubber stopper was placed in the other end to contain the carborundum, either tubing benders or conduit benders were used to put right angle bends in the large Cu tube in the appropriate places. Then the carborundum was removed and the tube cleaned. A hole is then drilled in the middle of the 19 mm Cu tube and a pump-out valve is silver soldered in place. A length of 5 mm (3/16”) outer

diameter hard Cu tubing typically 3000 mm long was selected and annealed to facilitate bending. Triangular spacers to hold the inner tube centered in the outer tube were cut from 1 to 2 mm (1/16") thick Teflon sheet with a hole punch. A hole was punched to accommodate the 5 mm tube and the circular Teflon was cut to a triangular shape with a razor blade. Some dozen or so of these spacers were suitably placed along the 5 mm Cu tube with a higher density of spacers in the region of the bends. These Teflon spacers were fixed in place with dabs of lead-tin solder applied to the 5 mm tube with a soldering gun. The tube was cleaned to remove the solder flux. One end of the 5 mm tube was flattened and a hole is drilled in the flat section so that a piano wire could be attached to facilitate pulling the 5 mm tube through the 19 mm tube. For this threading operation, the 19 mm tube was placed in a vise. Suitable bushings were then machined to allow the stainless steel vertical tubes to be silver-soldered in place for the vertical runs of the tube. The whole system is then cleaned to remove all the flux and the system can be leak-tested. Most transfer tubes had extenders that reached to the bottom of the helium dewar on one side and reached the bottom of the experimental cryostat on the other side.

Liquefiers in Physics Hall

Liquefaction of Nitrogen before 1967

In the late 1950s, two machines were purchased from the Joy Manufacturing Company to make liquid nitrogen, and they were located near the center of Room 0012 as shown in the sketch of Fig. 3. The Joy machines used ambient air and first made liquid air from which the 20% oxygen would be separated in a distillation column. This distillation process was efficient and generally trouble free, using the higher boiling point of oxygen of 90K compared to a boiling point of liquid nitrogen of 77K. To keep the machines from clogging with small amounts of water vapor and carbon dioxide, these gasses needed to be removed from the incoming air. To do this, the Joy machines used two reversing heat exchangers

that were alternated between a cold cycle operating at about 112K to trap the gasses and a warm cycle to purge the condensed carbon dioxide and water from the trap. The trap temperatures would be switched automatically. These Joy Liquefiers typically fed about 10 liters per hour into the 1000 liter storage tank just east of the liquefiers as shown in Fig. 3. In 1961, Sam Collins and R. E. Hughes of the Joy Manufacturing Company published the basic design ideas for these Joy Nitrogen Liquefiers in *Cryogenics.*[9]

Somewhat later, liquid nitrogen was purchased and stored in a 20,000 liter storage tank located at the warehouse near the railroad tracks. Commercial vendors supplied the liquid nitrogen and the Ames Laboratory truck would distribute it.

Liquefication of Hydrogen before 1967

A modified Collins liquefier was used to make liquid hydrogen. The Joule Thompson expansion valve fed into a closed tube and the cold helium gas was returned through the heat exchangers so that the machine was operated as a closed cycle helium refrigerator. Hydrogen gas from cylinders located along the north part of the west wall in Room 0012 was precooled with liquid nitrogen and fed into the Collins liquefier where it condensed on the cold end of the closed cycle helium refrigerator. As liquefied, this hydrogen was 75% ortho-hydrogen and 25% para-hydrogen, but it would transform over the course of a week or so to nearly pure para-hydrogen. Most of the liquid hydrogen was used by Spedding's group doing spectroscopic studies of rare earth insulating materials.[10]

Helium Liquefaction before 1967

Operation of the Collins helium liquefier required quite a bit of attention because helium was made in 8 to 10 liter batches in the Collins liquefier rather than in a large external dewar as is done now. In addition, the only place to store liquid helium was in 50 liter dewars that were

used by the experimental groups. Hence on any given morning, an inventory of the amount of helium on hand in these dewars would be made to plan the best time to start the liquefier. The operator would then check all the belts, the oil sump and the supply of He cylinders to be sure all was ready. The liquefier would be evacuated to remove any air in the system and then the system was purged three times with helium gas. The jacks that had been inserted under the valves at the time of the previous shut-down would be removed and the valve would be opened to start the liquid nitrogen precooling. About an hour and a half after the liquefier was started, one could expect to see liquid forming in the bottom of the Collins liquefier by looking through the sight glass at the top at the Styrofoam chunks floating on the liquid. After about 8 to 10 liters of liquid helium had been made, it would be transferred into the 50 liter storage vessels for use by the experimenters. Hence, helium transfers were needed about every hour.

The Move to Zaffarano Hall

In the early 1960s, there was a major nationwide push to rebuild the science infrastructure in the United States, driven partly by the Russian launch of Sputnick in 1956. Science education in the high schools was overhauled by the Physical Science Study Committee (PSSC). PSSC text books were written and PSSC physics summer schools for high school teachers were organized at Iowa State University. In addition, Dan Zaffarano was a member of an NSF Panel deciding which major universities might get new physics or science buildings. Dan, who was Chair of the Department at the time, organized the preparation of a proposal and Iowa State got a new Physics Addition that was finished in 1968. The National Science

Foundation paid half of the cost and the Iowa Board of Regents paid the other half. Later, the Physics Addition was named in honor of Dan Zaffarano.

Fig. 5 Zaffarano Hall under construction in 1967 and the front of Physics Hall from the south.

Zaffaranno Hall was a very sturdy construction that included a specification of 150 pounds/square foot of load bearing everywhere in the building so you could put a Varian iron core magnet anywhere in the building. On the north side of the building, the first floor rooms jutted out toward the north from the higher floors of the building providing wider rooms for the low temperature lab on the west end and the nuclear magnetic resonance labs on the east end of the building. This provided an extra-large contiguous space for the low temperature laboratory, and it provided a special room on the west end with a blow-out bubble for the hydrogen liquefier. There were other safety precautions for the use of liquid hydrogen in that the basement experimental rooms had explosion-proof lights and exhaust fans that were to be switched-on when liquid hydrogen was to be used. Alas, these facilities were rarely used because experimenters found ways to operate in the liquid hydrogen range using liquid helium as the coolant. The hydrogen liquefaction program was soon stopped.

Fig. 6 The 20,000 liter liquid nitrogen tank being taken to the warehouse and the same tank being reinstalled north of Zaffarano after the completion of the building.

About the time that construction of Zaffarano Hall was begun, liquid nitrogen was readily available commercially for about five cents a liter, so the Joy liquefaction machines were scrapped and a 20,0000 liter liquid nitrogen tank was purchased and located next to the Ames Lab receiving warehouse. During the construction period, Ames Lab trucks delivered 25 liter and 50 liter liquid nitrogen dewars to the low temperature lab in Zaffarano for dispersal to the research groups . About 1969, this 20,000 liter nitrogen storage tank was moved to the north side of Zaffarano and liquid nitrogen was dispensed to research groups from the north wall of the low temperature lab.

At the time of the move of the low temperature laboratory to Zaffarano Hall, a new Collins Model 200 liquefier was purchased and the first helium recovery lines were installed in chemistry, physics, Wilhelm Hall and Spedding Hall. These recovery lines were about 10 cm in diameter and were installed from the research laboratories to a central helium recovery system with banks of cylinders in the basement utilities room of Zaffarano Hall.

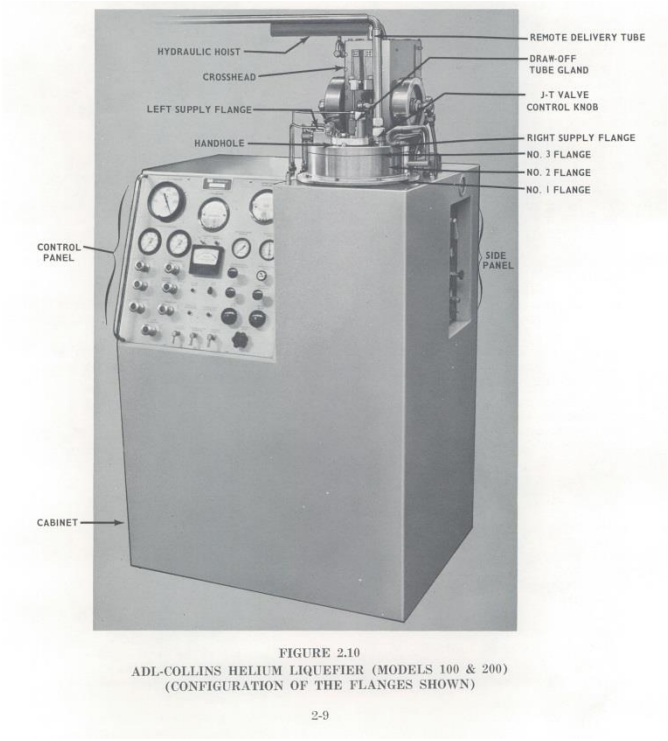

FIGURE 2.10
ADL-COLLINS HELIUM LIQUEFIER (MODELS 100 & 200)
(CONFIGURATION OF THE FLANGES SHOWN)

2-9

Fig. 7 ADL-Collins Helium Liquefier Model 200 as pictured in the instruction manuel.

Helium recovery and purification System

As originally installed in 1968, the helium recovery system was built with drain, waste and vent (DWV) grade plastic pipe with glued joints. At each station where liquid helium was transferred from a 50 liter dewar to an experimental dewar, there was a hook-up to the recovery system that included a plastic flow meter to measure the rate of flow of the returning helium gas. Other helium recovery stations were installed in the building hallways to collect blow-off while the dewars were stored between transfers. These flow meters were locally designed and constructed from a length of clear plastic placed vertically in the line and tapered over a length of about 15 cm so that a ping pong ball floated in the gas stream to indicate the

rate of flow. Spedding, Wilhelm and Zaffarano Halls as well as many areas of chemistry were served by the original recovery system. Molecular Biology Building was added in 1992 and Hach Hall was connected when it opened in 2010. Over the years, the plastic recovery lines were replaced with copper because repeated freezing caused cracks in the plastic line.

Liquid hydrogen was used for only a short time after the move to Zaffarano Hall. Spedding retired in 1973 and there was increasing pressure from safety personnel to limit the use of liquid hydrogen. Hence, sometime in the early 1970s, the hydrogen liquefier was removed and that northwest room of the low temperature laboratory became devoted to compressors for helium recovery and purifier cabinet. A large gas bag and bank of about 90 helium cylinders for storage were put in the northwest end of the utility room in the basement. Over the years the number of storage cylinders grew to several hundred.

Operation of the ADL-Collins Liquefier-Model 200

In 1968, an ADL-Collins Liquefier-Model 200 was purchased and installed in Zaffarano Hall. It had an external Joule-Thompson valve and delivered liquid directly into a 500 liter dewar so that liquid could be produced over night without attention. This liquefier was more automated than previous models but considerable attention still was needed to keep it running. Typically it delivered liquid at a rate of 10 to 12 liters per hour. The layout of the laboratory shown in Fig. 7 has the liquefier in the south room and two stacks of helium compressors across the wall in the north room. The 1000 liter nitrogen storage tank was just west of the compressors and

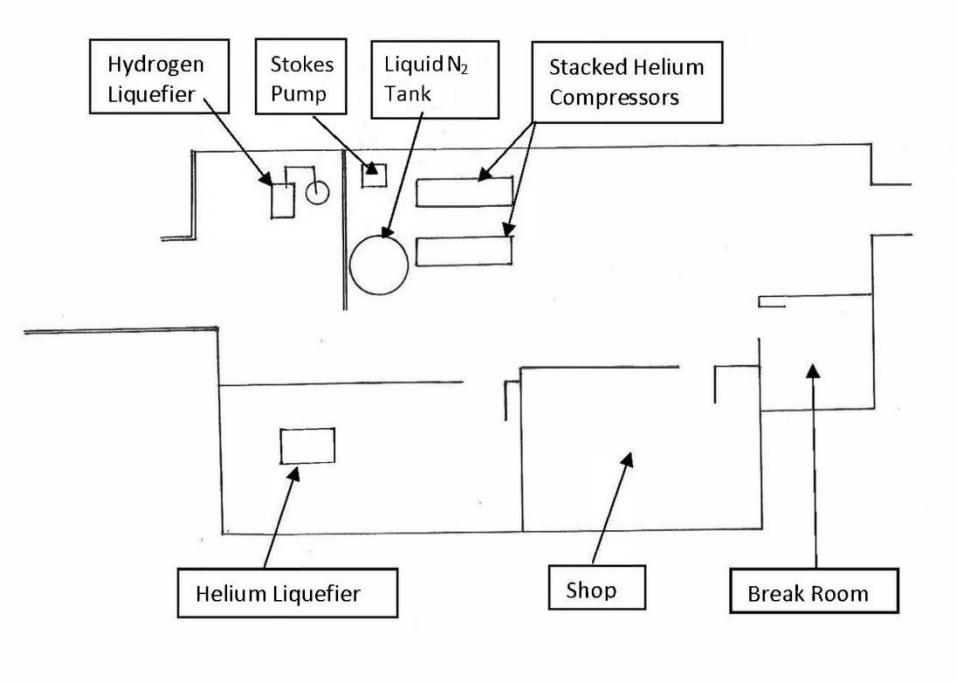

Fig. 8 Sketch of the Low Temperature Laboratory in Zaffarano in 1968.

there was a Stokes pump for large volume evacuations when needed. Typically, the storage of 100 liter or 60 liters helium storage tanks for research groups was along the north wall.

Fig. 9 Helium storage along the north wall

Two stacks of helium compressors with two helium compressors each were available to serve the helium liquefiers, but only one pair would be used at a time. The other pair would be a back-up. As a cost saving measure, Ames Lab continued to use reciprocating compressors for a long time after other labs were going to screw compressors. Ed Wolf was a new faculty member at that time in 1976 and he helped us start using the Department of Energy excess-equipment list for equipment that some other national lab had purchased but was not going to use because of changing priorities. Ed obtained several items of high vacuum equipment that often were new. From the same lists we found excess helium liquefier equipment. Mike Sandholm drove a truck to University of Illinois at Urbana-Champaign to pick up two helium compressors and Jerry Ostenson went to Puerto Rico and arranged to have two more used compressors and other liquefier equipment shipped to Ames. This led to considerable time repairing old compressors.

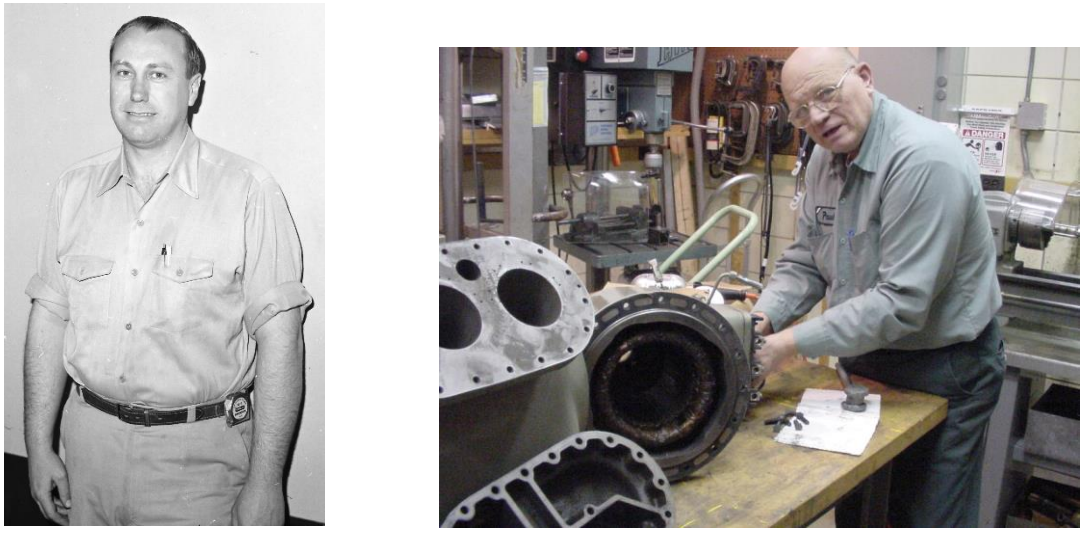

Fig. 10 Mike Sandholm on the left and Paul Ness repairing a reciprocating helium compressor in the shop on the right.

Operation of the Model 1410 Liquefier

In 1997, the model 1410 helium liquefier was purchased from Process Systems International (PSI) as shown in Fig. 11. For this liquefier, a helium gas purifier is built into the machine and the production rate runs consistently over 20 liters per hour.

Fig. 11 The model 1410 helium liquefier with the top panels off and a 600 liter storage dewar. At the right is Marc McGinn with the Model 1410 liquefier with the panels in place.

Work on the Temperature Scale

Clayton Swenson had a long interest in the accuracy of the temperature scale and focused substantial effort toward increasing both the accuracy and the precision of measurements across the lab. While he was still at MIT he teamed up with Bobby Berman, who was on leave from Oxford, to determine the vapor pressure of liquid helium from 4.2K to 5.2K using constant volume gas thermometry. They found differences from the then currently accepted scale of about 20 millikelvin.[11] We then used constant volume gas thermometry to calibrate some germanium resistors for specific heat work to 20K and found[12] the vapor pressure of equilibrium hydrogen at a saturated vapor pressure of 760.00 Torr to be 20.253K

plus or minus 0.03K, a value intermediate between other measurements at the time.[12] We also calibrated some Au—0.07 at% Fe thermocouple wire to use as a secondary thermometer in the 4 to 20K range.[13]

For some time, there had been evidence that the accepted temperature scale called T_{58} was somewhat in error. In 1955, the Westinghouse group had measured the specific heat of copper, silver and gold from 1 to 5K and found good agreement with theory if the temperature scale was adjusted by amounts in the ten millikelvin range.[14] Then in 1970, Tom Cetas' thesis used paramagnetic salt magnetic thermometry to show that the boiling point of helium given by T_{58} was too low by about 7 millikelvin.[15] These data were not definitive, but they clearly pointed to a need for improvement in the T_{58} scale. This launched Swenson into a mini-career working on the Consultative Committee on Thermometry, International Commission on Weights and Measures for many years to develop an improved temperature scale.

Germanium resistance thermometers were the work-horse instrument for determining and transferring temperature. Swenson's group constructed a germanium resistance comparator apparatus so that as many as a dozen new germanium thermometers could be calibrated against a standard at the same time. It was a large cylindrical copper block with cylindrical holes to make good thermal contact to the germanium and multiple long wire lead wires that were well anchored thermally to the copper block to intercept any heat coming to the resistor. With these calibrations, we could provide a thermometer to any group that wanted to use a scale that we thought was the best approximation to an international scale.

A few parting words

There were a couple of other experiments that were not directly related to the Low Temperature Laboratory that might be mentioned. John Wollan built the first ^{3}He – ^{4}He dilution refrigerator in Ames, Fig. 12, using sintered copper heat exchangers. It reached 60 millikelvin.

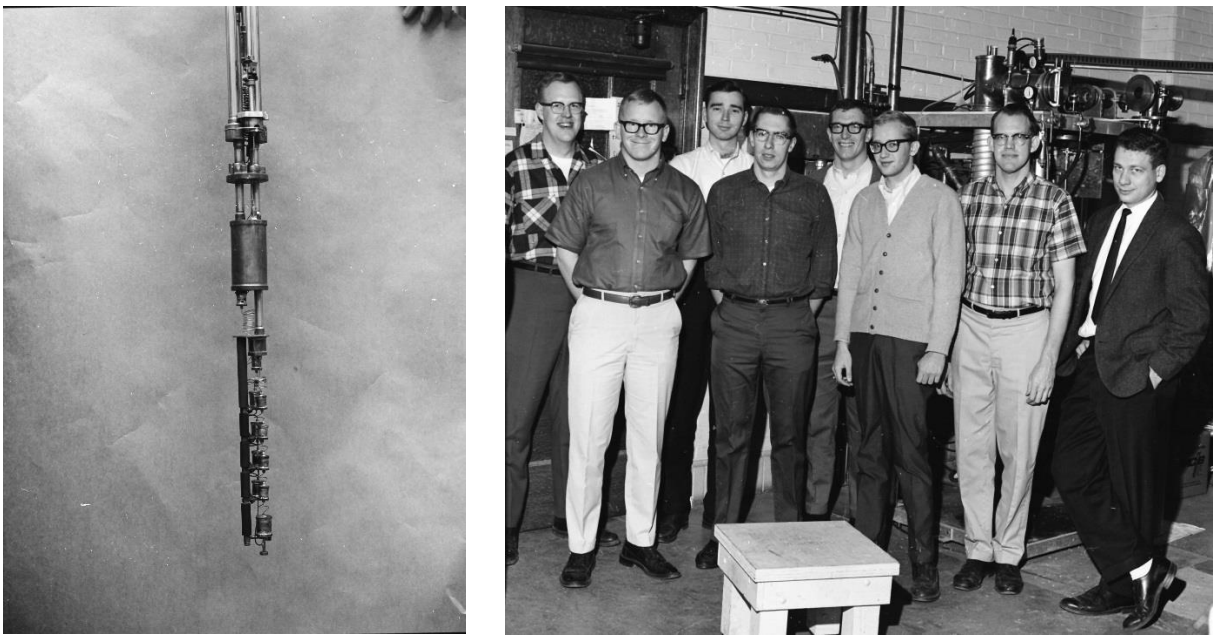

Fig. 12 John Wollan's dilution fridge shownat the left. At the right is a 1966 photo of the Finnemore group with Jerry Ostenson, Rick Rump, John Wollan, Brent Haskell, Larry Williams, Bob Jones, Wayne Decker and Doug Finnemore, left to right.

A one of a kind experiment to grow free standing rare gas solids and measure their thermal expansion was carried out by Charles Tilford in Swenson's group. He first grew solid Argon in the apparatus shown in Fig. 13 and measured the thermal expansion coefficient from 1 to 25K.[16] He continued to measure krypton and xenon. In the midst of the construction of Zaffarano Hall, Charles also grew single crystals of solid helium at the reactor for the first measurement of the truly soft phonon modes of helium by Sunil Sinha.[17]

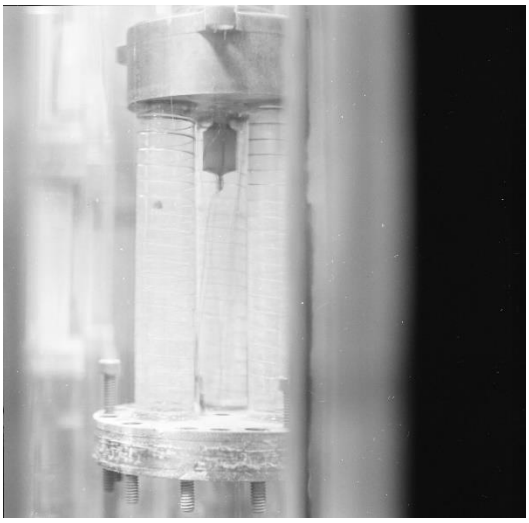

Fig. 13 The photo to the right shows Swenson's group in 1967 with the back row being Marv Anderson, unknown, Charles Tilford, Bob Fugate (of adaptive optics fame), Tom Cetas and Clayton Swenson. The bottom row shows unknown, Kent McLean and Paul Sparks.

Acknowledgements – Jerry Ostenson and Paul Ness made major contributions to this document, providing ideas and basic facts as well as adding to the overall story. Clayton Swenson was a key source for events before 1960 and both the Ames Lab and Departmental staffs were very helpful. Steve Karsjen and Kerry Gibson provided many old photographs, and Rolf Hansen provided new ones.

References

1.) D. H. Parkinson, F. E. Simon and F. H. Spedding, Proc. Roy. Soc. (London) 207, 856 (1952)

2) N. R. James, S. Legvold and F. H. Spedding, Phys. Rev. **88,** 1092 (1952)

3) J. F. Elliott, S. Legvold and F. H. Spedding, Phys. Rev. **91,** 28 (1953)

4) J. F. Elliott, S. Legvold and F. H. Spedding, Phys. Rev. **94,** 50 (1954)

5) M. Griffel and R. E. Skochdopole, J. Amer. Chem. Soc. **75,** 5250 (1953) and M. Griffel, R. E. Skochdopole and F. H. Spedding, Phys. Rev. **93,** 657 (1954).

6) Private communication B. C. Gerstein.

7) F. E. Simon and C. A. Swenson, Nature, **165,** 829 (1950); C. A. Swenson, Phys. Rev., **79,** 626 (1950); C. A. Swenson, Phys. Rev. **86,** 870 (1952).

8) Private communication with C. A. Swenson.

9) S. Collins and R. W. Hughes, Cryogenics, **2,** 43 (1961).

10) W. J. Haas, R. W. G. Syme, R. H. Good Jr., **47,** 1572 (1967).

11) R. Berman and C. A. Swenson, Phys. Rev. **95**, 311 (1954).

12) D. K. Finnemore, D. L. Johnson, J. E. Ostenson, F. H. Spedding and B. J. Beaudry, Phys. Rev. **137,** A550 (1965).

13) D. K. Finnemore, J. E. Ostenson and T. F. Stromberg, Rev. Sci. Inst. **36,** 1369 (1965).

14) W. S. Corak, M. P. Garfunkel, C. B. Satterthwaite, and A. Wexler, Phys. Rev. **98,** 1699 (1955).

15) T. C. Cetas and C. A. Swenson, Phys. Rev. Lett., **25,** 338 (1970).

16) C. R. Tilford and C. A. Swenson, Phys. Rev. Lett. **22,** 1296 (1969).

17) T. O. Brun, S. K. Sinha, C. A. Swenson, and C. R. Tilford, *Neutron Inelastic Scattering* (International Atomic Energy Agency, Vienna, 1968) Vol 1, p. 339.

Personnel of the Low Temperature Cryogenic laboratory

Ralph Houchin (retired 1961)

Paul Ness (1962-2012)

Mike Sandholm (1961-2001)

Marc McGinn (since 2001, retirement 2018)

Bill Wing (2013-2015)

Robert Howard (March 2015 current).

Nick Kriegel (January 2018)

Supervisors

Jerry Ostenson (1964-1997)

Sergey Bud'ko (1997-2014)

Makariy Tanatar (since November 2014).

Key Professors

Sam Legvold

Clayton Swenson

Doug Finnemore