Information Circular 9442

Cryogenics Demonstrations

By Samuel R. M. Burton

UNITED STATES DEPARTMENT OF THE INTERIOR
Bruce Babbitt, Secretary

BUREAU OF MINES
Rhea Lydia Graham, Director

CONTENTS

ILLUSTRATIONS

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

in	inches	psig	pounds per square inch, gauge
K	kelvin	°C	degree Celsius
kg	kilogram	°F	degree Fahrenheit
mm	millimeter	%	percent
ppm	part per million		

CRYOGENICS DEMONSTRATION

By Samuel R. M. Burton

ABSTRACT

Presentations are given to disseminate information about helium and cryogenic effects on common materials. This document provides a standard of content for cryogenic experiments and lists equipment utilized during U.S. Bureau of Mines (USBM) cryogenics demonstrations. The demonstrations use liquid nitrogen to obtain the cryogenic temperatures.

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INTRODUCTION

As a public service, USBM provides technical information about helium production. Numerous requests by teachers, students, and other USBM centers for information on the USBM cryogenics demonstrations can now be met. The effects of cryogens on materials used in the production of helium and transportation of gaseous helium are given. This information is provided to local schools, civic organizations, and the public with cryogenic demonstrations. During the demonstrations, changes in properties, change of state, and the effects of the cryogenic temperatures on common materials at liquid nitrogen temperature (-195.8 °C, 77 K) are observed.

CRYOGENICS DEMONSTRATIONS

Temperature scales are useful to better understand the change of properties from room temperature to the cryogenic region. The Celsius scale, shown in figure 1, is generally used for scientific temperature measurements, and the Fahrenheit scale is the most common temperature scale used in the United States. Some temperatures (*6*) of interest are: boiling point of water 100 °C (212 °F), freezing point of water 0 °C (32 °F), mercury solidifies at -38.9 °C (-38.0 °F), carbon dioxide sublimes at a temperature of -78.5 °C (-109.3 °F), and the coldest temperature measured in nature was -89.2 °C (-128.6 °F) at the former Soviet Union's Vostok station in Antarctica on July 21, 1983 (*2*). The liquefaction temperatures of some common gases are: methane -164.0 °C (-262.3 °F), oxygen -183.0 °C (-297.4 °F), air about -190 °C (-310 °F), nitrogen -195.8 °C (-320.4 °F), hydrogen -252.9 °C (-423.2 °F), and helium -268.9 °C (-452.1 °F). The reference point is absolute zero at -273.15 °C (-459.67 °F). A comparison of various cryogens is given in figure 2.

HELIUM LIFTING CAPACITY AND PERMEABILITY

A cylinder of helium is shown in figure 3. Compressed helium from the cylinder is expanded to inflate a rubber balloon. The lifting capacity of helium is about 1 ounce per cubic foot. The weight of the balloon is less than 1 ounce and as helium fills the balloon the lifting capacity of the helium is greater than the weight of the balloon, and the balloon floats.

Helium diffuses through the rubber balloon, and since the concentration in the balloon (100%) is greater than the helium in the air (5 ppm), more helium will diffuse out of the balloon than into the balloon (*3*). Within 2 to 3 days, the weight of the balloon is more than the lifting capacity of the helium left in the balloon, and the balloon will descend to the floor. In the case of a mylar balloon, it may take weeks instead of days.

To demonstrate helium leak detection, a helium-filled balloon and a commercial helium leak detector are used. The leak detector is an analytical instrument called a mass spectrometer that is tuned for detection of traces of helium-4. The helium that diffuses through the balloon is sampled and the instrument will indicate the presence of helium by a lighted bar graph. As the concentration of helium in the air increases, the length of the lighted bar graph increases.

To assist the detection of the helium diffusing through the balloon, the palm of the hand is placed on the balloon and the sniffer probe placed under the hand. The helium leak detector quickly indicates the concentration of helium. By using a balloon made of Mylar, a significant decrease in the leak rate can be demonstrated. This decreased rate explains the increased length of time a Mylar balloon stays inflated.

Mass spectrometer-helium leak detectors are used in laboratories, helium plants, and gasfields as a means of finding leaks in pressurized helium pipelines and other pressurized or vacuum apparatus.

Permeability of helium through a porous ceramic crucible can be demonstrated with an inverted beaker filled with helium and a Magnehelic pressure gauge, as shown in figure 4. The inverted beaker filled with helium is placed over the crucible. Helium diffusing through the crucible causes increased pressure on one side of the diaphragm in the Magnehelic gauge and is indicated by the movement of the needle on the gauge.

Two core samples, one of anhydrite and another of brown dolomite, are passed around in the audience. These materials were taken from the geologic formations where helium is stored. Helium is found in the natural gases that are stored in the brown dolomite (that has a porosity of 10% (*8*)) at Cliffside Gas Field. The anhydrite is representative of the rock over the gas field and does not allow the gas to escape.

CHANGES IN PHYSICAL PROPERTIES AT LIQUID NITROGEN TEMPERATURES

Liquid nitrogen (N_2) is used as a refrigerant in the cryogenic process to separate helium from natural gas.

Dewars, as shown in figure 5, are used as containers for liquid nitrogen. These dewars are similar to the thermos bottles used in lunch boxes, except they are made of Pyrex glass, have a low coefficient of expansion, and can withstand greater changes of temperature. Liquid nitrogen is colorless and odorless. When it is poured into a dewar, vigorous boiling takes place. This boiling is the result of heat being removed from the container, changing liquid nitrogen to gaseous nitrogen. After the heat is removed and the container has reached liquid N_2 temperature, the boiling subsides.

Liquid nitrogen is used to show physical property changes at cryogenic temperatures. A hollow rubber racquetball placed in liquid nitrogen becomes rigid. The air inside the hollow ball liquefies, forming a partial vacuum. When the frozen ball is thrown against a hard surface, it shatters, as shown in figure 6. The noise made as the ball shatters is an implosion, caused by the inward bursting of the ball.

When a piece of rubber tubing is stretched and released at room temperature, it returns to its original shape. When the tubing is immersed in liquid nitrogen, vigorous boiling indicates that heat is being removed from the rubber tubing. After the boiling subsides, the rubber tubing has reached liquid N_2 temperature and is rigid, as shown in figure 7. At this temperature its properties have changed and when pressure is applied to bend the tubing it breaks. When the rubber warms to room temperature, it becomes elastic again.

Because of the cryogenic temperatures used during the purification and liquefication of helium, care is required in the selection of materials that will be used in the construction and modification of helium plants. Common steel can become brittle and shatter at liquid nitrogen and liquid helium temperatures. Valve packing also requires careful selection, since the rubber "O" rings used at normal temperatures will harden at cryogenic temperatures.

The effect on metal tubing is demonstrated using the apparatus shown in figure 8. Stainless steel tubing and common steel conduit, of the same internal diameter (ID) and outer diameter (OD), are cooled to liquid N_2 temperatures and tested with the impact delivered from a 2.4 Kg weight dropped 32 inches. The demonstration shows that stainless steel, when cooled to liquid nitrogen temperature, will deform when struck but not shatter, whereas the common steel shatters.

The properties of organic materials also change at cryogenic temperatures. After a few minutes in liquid nitrogen, a banana becomes hard enough to be used to drive a nail into wood, as shown in figure 9. Another example of an organic property change is freezing a flower. The frozen flower will shatter when struck against a hard surface.

CHANGES IN VOLUME AND STATE

As liquid nitrogen is warmed, it changes to a gas. Nitrogen at room temperature and atmospheric pressure, occupies about 710 times more volume than when liquid (*7*). When storing or transporting liquid nitrogen, pressure relief of the container is essential. To demonstrate this change of state, liquid nitrogen is poured into an Erlenmeyer flask. A balloon is placed over the neck of the flask, and the gaseous nitrogen will be contained at about atmospheric pressure, as shown in figure 10. The boiloff rate of the liquid nitrogen is proportional to the amount of heat absorbed by the liquid nitrogen.

A balloon inflated with air is used to demonstrate the reduction in volume that occurs when air is cooled with liquid nitrogen. Liquid nitrogen is poured over the balloon and the balloon shrinks, as shown in figure 11. By using a transparent balloon, you can see that carbon dioxide and water have been solidified forming a thin film on the interior surface. When the balloon is moved through the air, it will warm and return to its original size. This also demonstrates the expansion in volume that occurs when air is warmed.

ELECTRICAL RESISTANCE

The resistance to electrical current in metals decreases at cryogenic temperatures (*1*). A single cell flashlight with a coil of copper wire in series with a light bulb and battery is used to demonstrate this change. When the coil of copper wire is cooled to the temperature of liquid nitrogen, the brightness of the light increases as the resistance of the copper wire is reduced. This is a result of improved electrical conductivity through the cryogenically cooled copper wire.

CRYOPUMPING

Another use for cryogenic liquids is cryopumping (*4*). Cryopumping occurs when gas molecules are condensed, or trapped on a low-temperature surface. A cryophorus, shown in figure 12, is used to demonstrate this phenomenon. Distilled water was placed in the cryophorus (a drop of red food coloring is added for visibility), the gas above the water was removed, and then the cryophorus was sealed. Therefore, the cryophorus contains water and water vapor. The water maintains an equilibrium with the water vapor at the temperature of the cryophorus. To demonstrate cryopumping, the liquid water is moved into the bulb and the long leg of the cryophorus-containing water vapor is immersed in liquid N_2. At liquid nitrogen temperature, the water vapor in the leg will solidify. This reduces the pressure above the liquid, and the liquid water will vaporize to supply the vapor pressure required at room temperature. As the liquid water vaporizes enough heat is removed by the heat of vaporization to freeze the surface of the water, as shown in figure 12. Three states of matter (solid, liquid, and gas) are present in the cryophorus. When the cryophorus is removed from the liquid nitrogen, the ice on the surface of the water is melted by rubbing the bulb to add heat. Cryogenic distillation is also observed, because only water is vaporized and frozen in the low temperature area. The ice will melt to form clear water that is free of food color.

CHANGE OF STATE FOR CARBON DIOXIDE

Carbon dioxide is used to demonstrate a change of state from a gas directly to a solid. A small balloon is filled with carbon dioxide and placed over a test tube. The test tube is immersed in liquid nitrogen, and the gas that is in contact with the low temperature is changed to a white solid by cryopumping, as shown in figure 13. This material is commonly called "dry ice," which is commercially manufactured for use as a refrigerant. After the test tube is removed from the liquid nitrogen, the test tube will warm and the "dry ice" will return to the gaseous state.

LIQUEFYING AIR AT LIQUID NITROGEN TEMPERATURES

To liquefy air, about 1/2 liter of liquid nitrogen is poured into a copper-bottomed teakettle. After a few seconds, droplets of liquid air form and run off the bottom of the teakettle, as shown in figure 14. The liquid nitrogen inside the teakettle is at a temperature of about -195.8 °C, and air liquefies at a temperature of about -190 °C.

PRESENTATION GUIDELINES

These educational cryogenics demonstrations have been presented for over 35 years with annual attendance estimated at 800-1,000 persons per year. When planning to give this demonstration, personnel should be checked out in the use of the demonstration equipment and adherence to the following guidelines:

1. Visual inspection of all equipment before use.
2. Amount of liquid nitrogen required.
3. Use only approved apparatus and procedures.
4. Audience must be at a safe distance from the demonstrations.
5. Audience does not participate.
6. Safe handling of cryogenic liquids.
7. Safe disposal of excess liquid nitrogen.
8. Know dangers of pyrex glass dewars and metal dewars.
9. Use of only pure liquid nitrogen.

All presentations are different, due to the type of audience and individual style of the presenter, but must be technically correct. The equipment checklist, Appendix A, and the general outline in Appendix B should assist in preparing for the demonstration. A list of most frequently asked questions can be found in Appendix D. Questions about sales and users can be answered using information in Appendix E and F.

CONCLUSION

The USBM provides cryogenics demonstrations to disseminate information about helium and cryogenic effects on common materials. These demonstrations offer educational opportunities and hopefully will ignite an interest in pursuing the study of the sciences. The presentations are approximately 45 minutes to an hour in length and are given to an estimated 800-1,000 persons annually.

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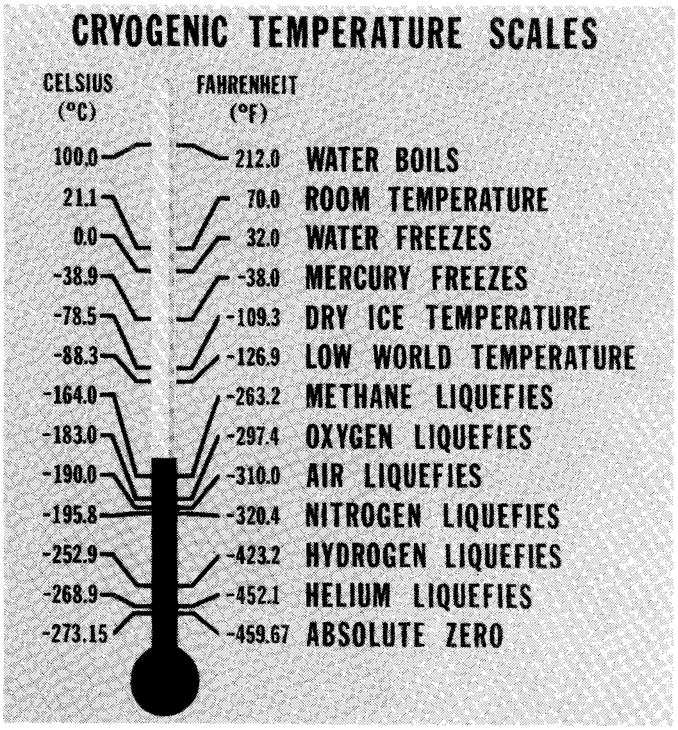
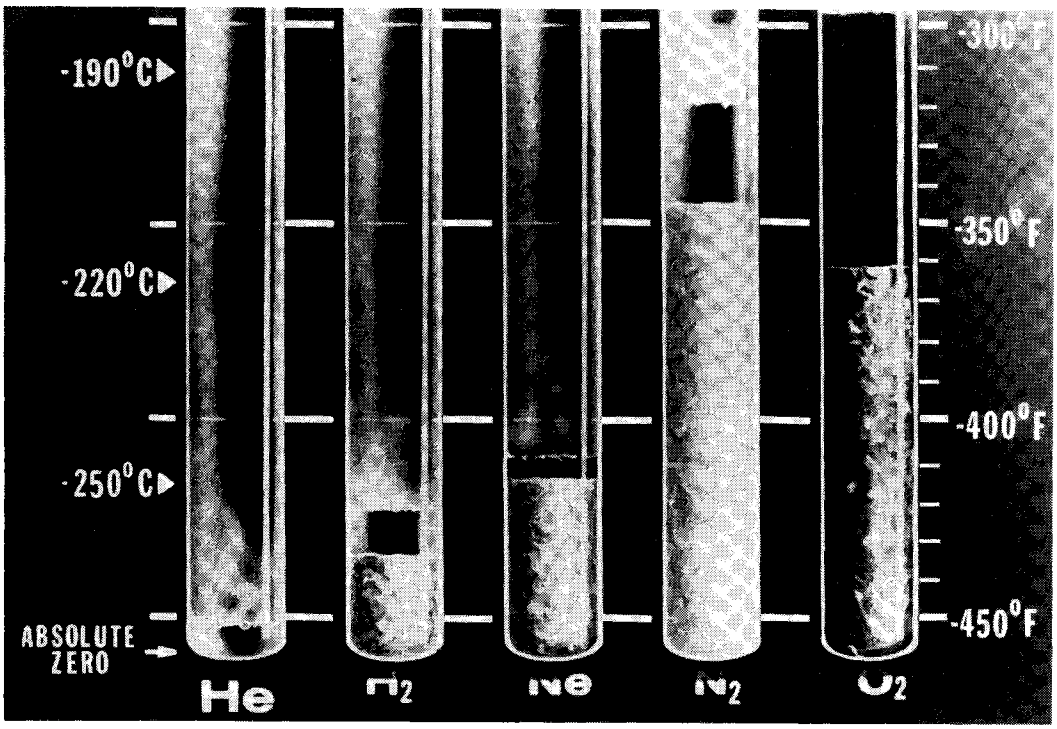
Figure 1



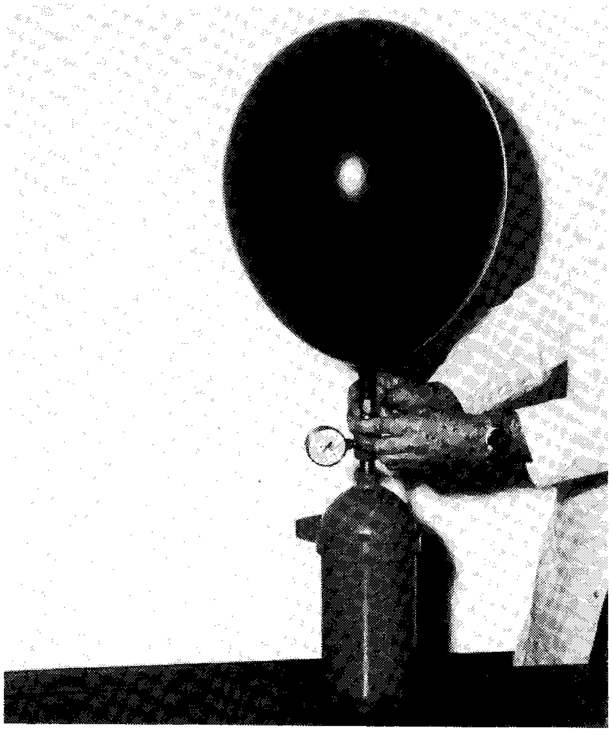
Cryogenic temperature scales.

Figure 2



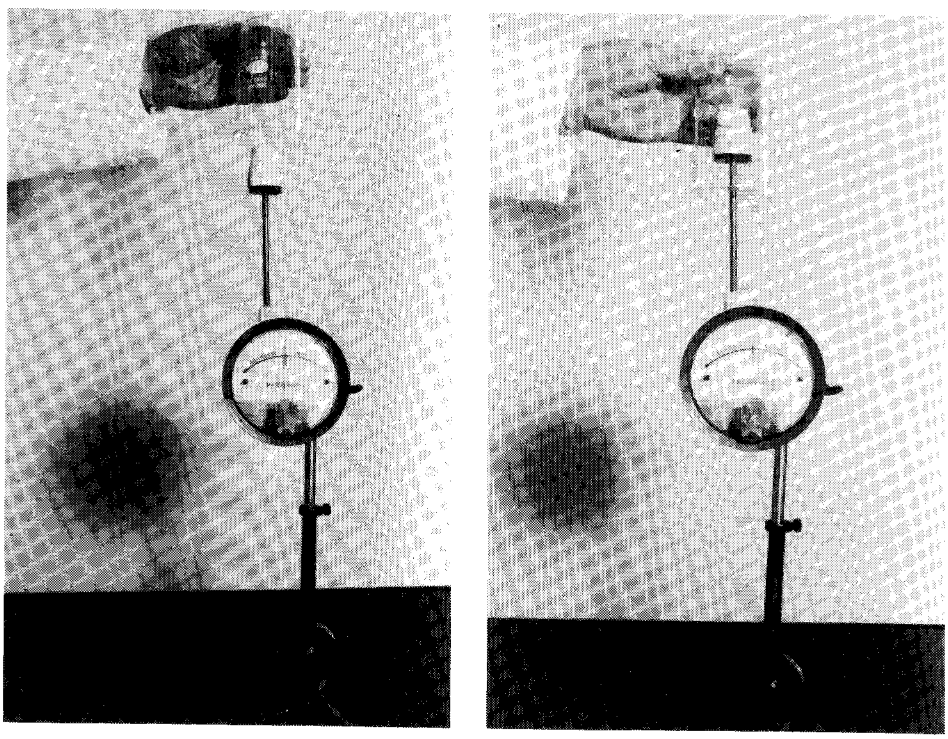
Liquefaction and solidification temperatures of cryogens.

Figure 3



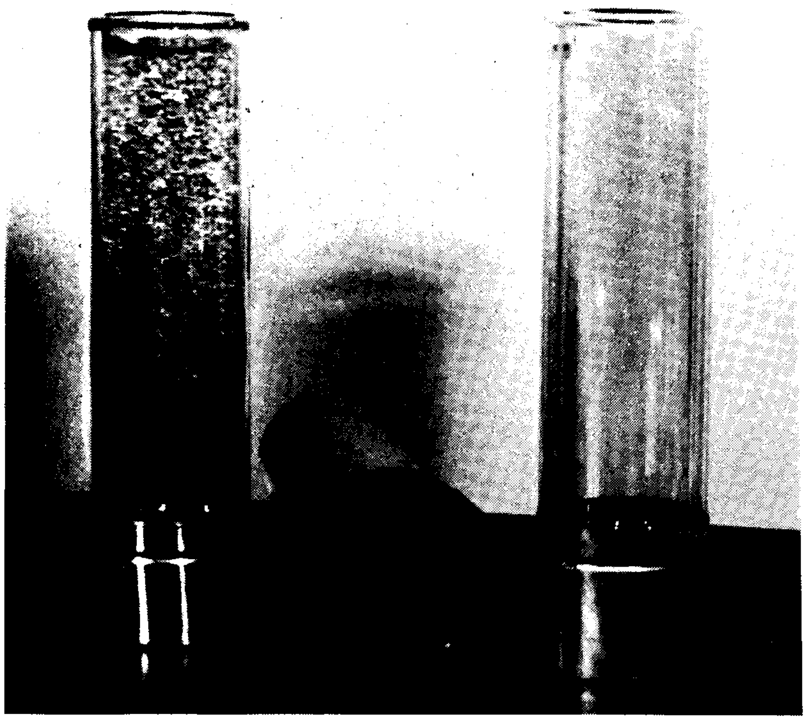
Lifting capacity of helium.

Figure 4



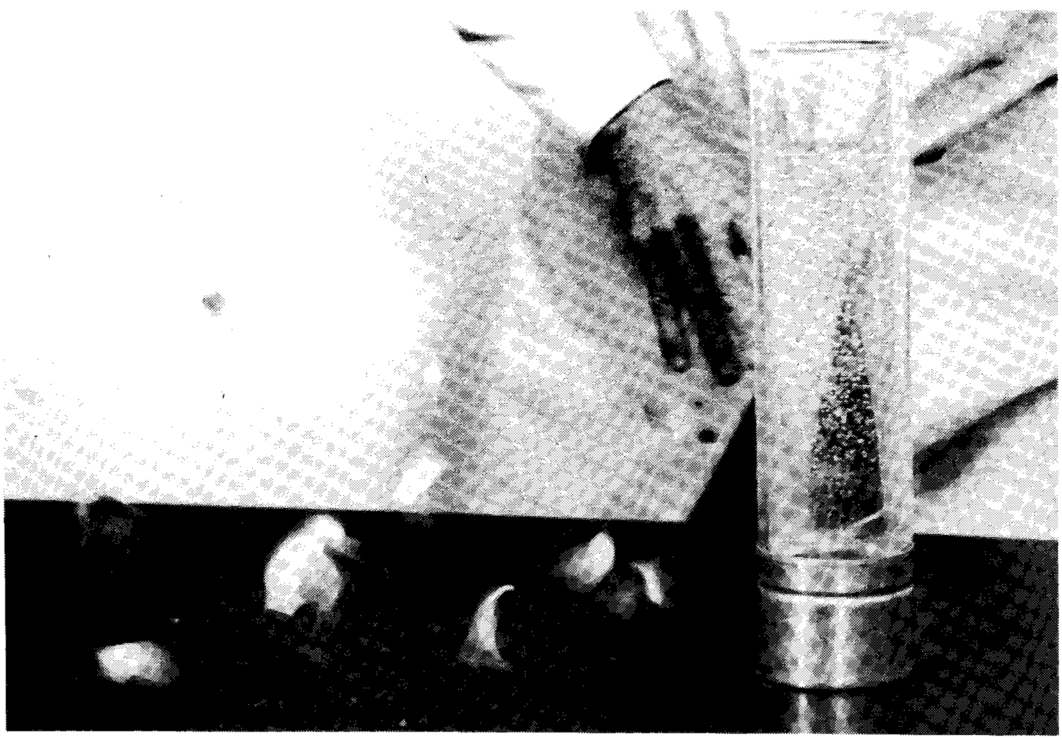
Magnehelic pressure gauge demonstration.

Figure 5



Dewars for cryogenic liquids.

Figure 6

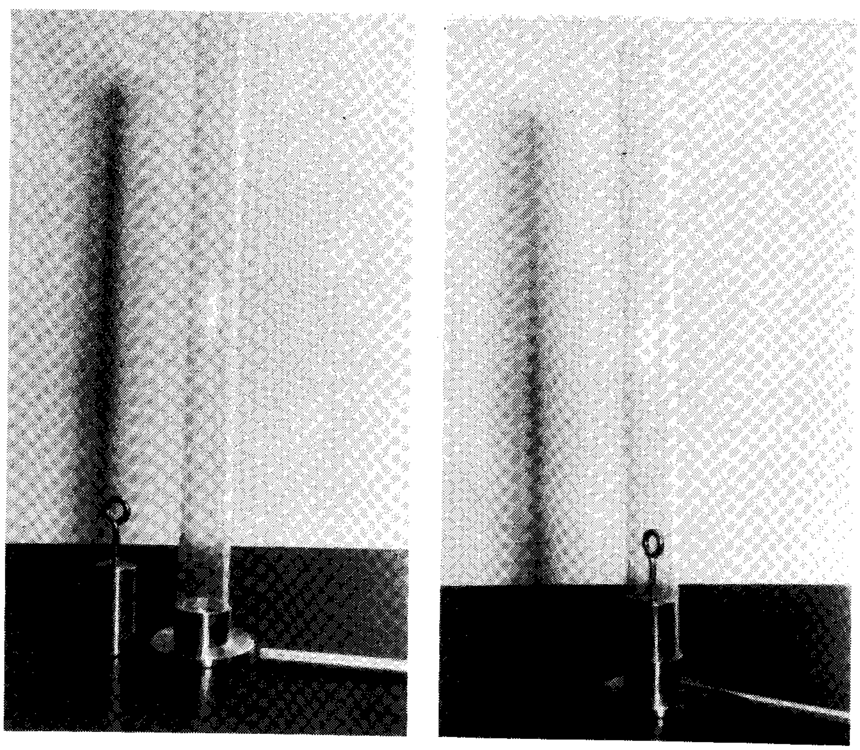


Shattered rubber ball.

Figure 7

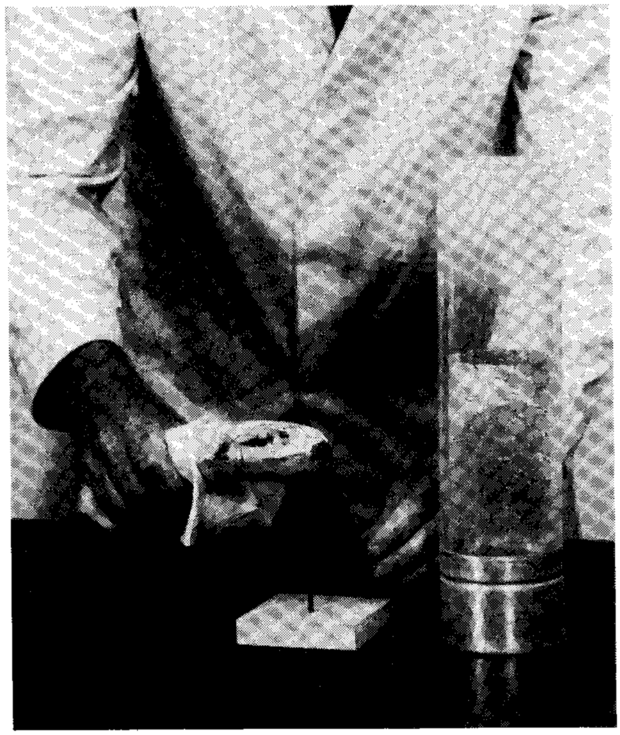
Rubber tubing at liquid nitrogen temperature.

Figure 8



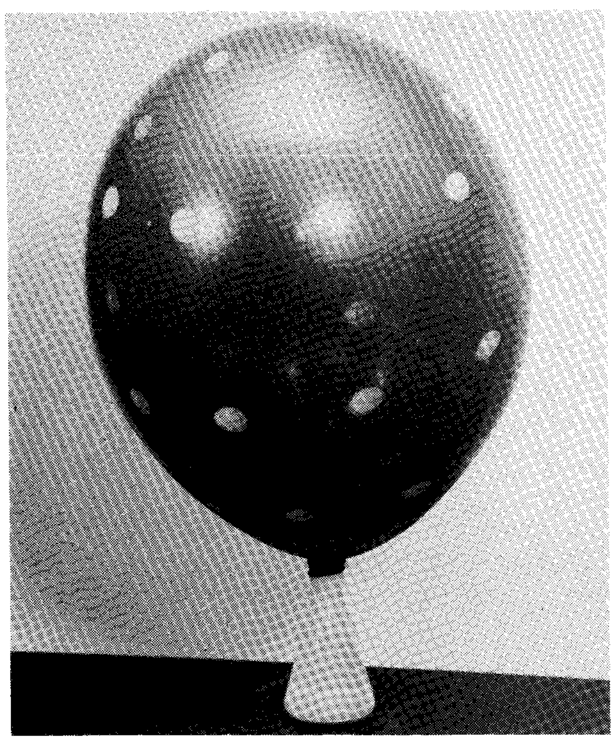
Metal tubing impact testing apparatus.

Figure 9



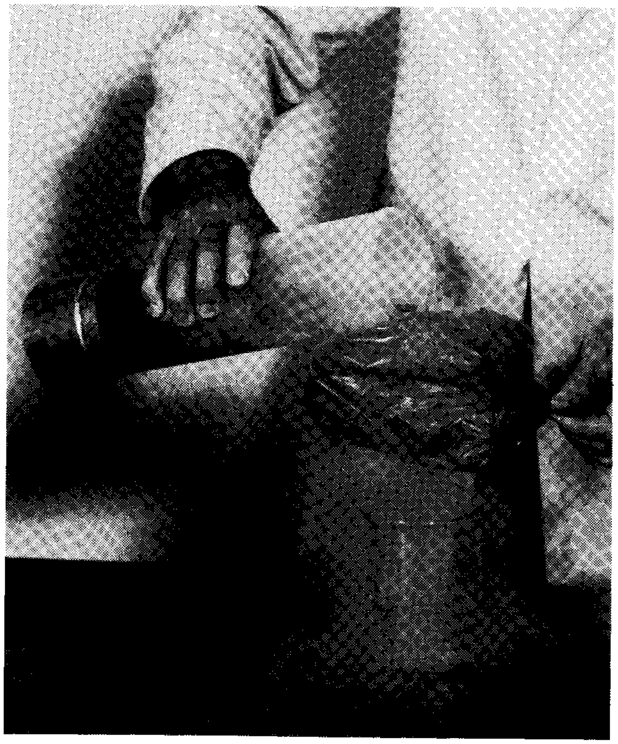
Driving a nail with a frozen banana.

Figure 10



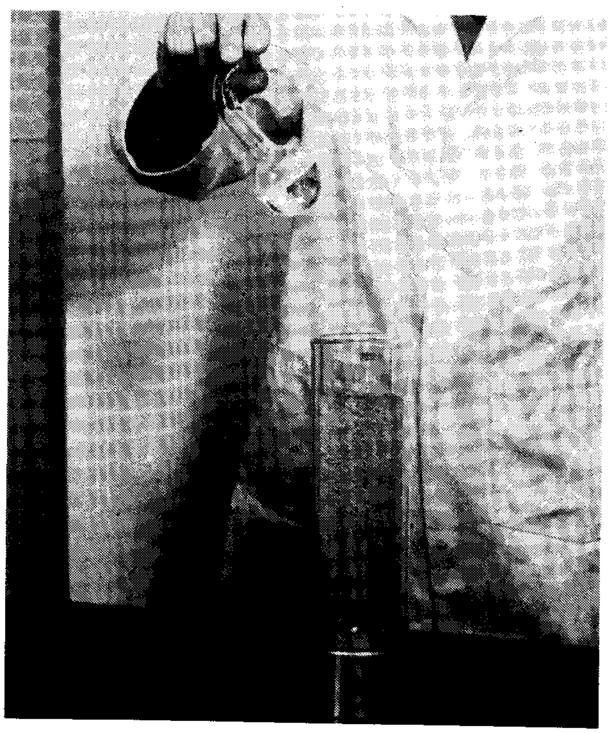
Change of volume from liquid N_2 to gaseous N_2.

Figure 11



Change of air volume.

Figure 12

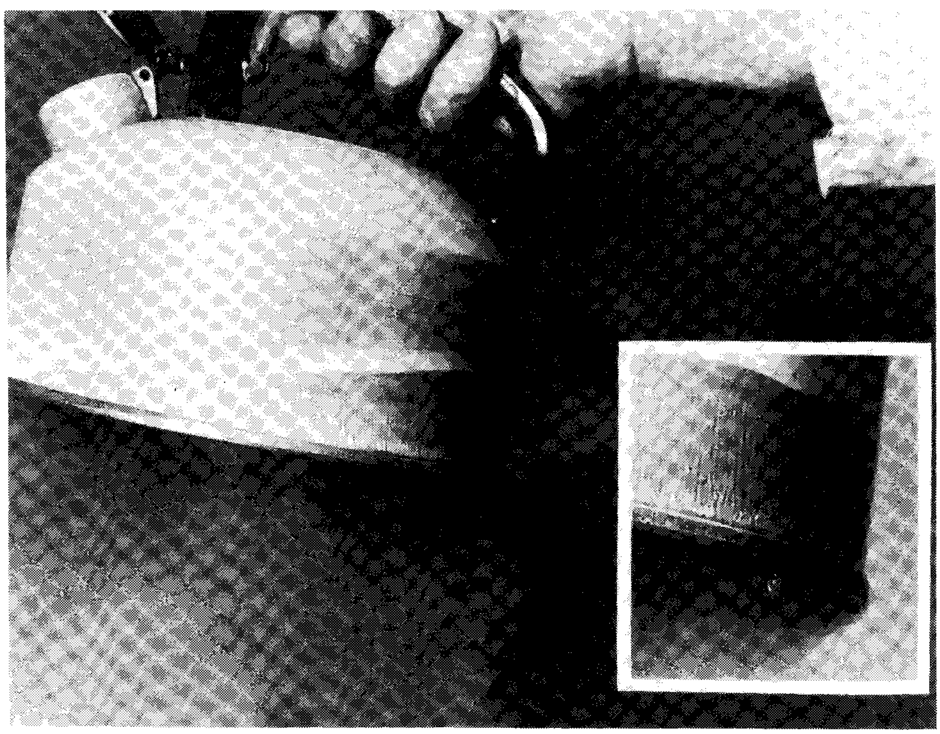


Cryophorus.

Figure 13

Cryopumping carbon dioxide.

Figure 14



Liquefying air at liquid nitrogen temperature.

APPENDIX A

Equipment Checklist for Cryogenics Demonstration

		Check if Present
Authorization to present information to the public (See Appendix F for example)	(mandatory)	______
Thermal expansion apparatus	(optional)	______
(Plexiglass) metal tubing impact testing apparatus	(optional)	______
Digital LED resistance indicator	(optional)	______
Mylar balloon	(optional)	______
Mass spectrometer leak detector	(optional)	______

- "A" Kit -

Rubber balloons (test for holes before use)	______
Corks	______
Goggles, safety/full face shield	______
Leather gloves	______
Teakettle	______
Pointer	______
Rubber tubing	______
1 Erlenmeyer flask	______
Glass dewar	______
Conductivity flashlight (check battery)	______

- "B" Kit -

Paper towels	______
Extension cord	______
String for balloons	______
Scissors	______
Rubber balls	______
Wooden block	______
CO_2 cylinder	______
Tongs	______
Diffusion apparatus (MAGNEHELIC gauge) and beaker	______
Core samples	______
Cryophorus	______
Hammer	______
Nail	______
Lead bell	______
Pyrex test tube and balloon for CO_2 sublimation	______
Banana (needed)	______
Flower (needed)	______

Other Apparatus

Helium cylinder (100 psig minimum)	______
Posterboards and poster stand	______
10 Liters of liquid N_2	______
2 Audience shields	______
Speaker system and tie-clip microphone (optional)	______
Cryogenic temperature thermometer (Poster on stand)	______
Overhead projector (optional)	______
Slide projector (optional)	______

APPENDIX B

Recommended Outline for Cryogenics Demonstrations

Before the demonstration, call the organization/club organizer to confirm location, date, allotted time, and expected attendance. Ask if electrical power is available for the speaker system (overhead and slide projector) and if full-size tables will be provided. Before starting your presentation, display the Gelb diagram (5) (Appendix C) to your audience. This will allow them to organize your presentation and remember what you have told them. Review the following outline:

1. Introduction (Organization, you, and your partner).

   a. Be brief.
   b. Know your outline.

2. Federal Helium Program - Provides helium to Government agencies.
3. Headquarters in Amarillo (approximately 180 employees). The helium time column is located at the Discovery Center. The 25-year time capsule was opened May 15, 1993.
4. Cliffside Gas Field - 12 miles north of Amarillo, containing about 35 billion cubic feet of Government-owned helium. Gas is moved by pipeline.
5. Amarillo Helium Plant (built in 1929) - 2 miles west of Amarillo at the intersection of Amarillo Boulevard and Helium Road. This plant operates a cylinder- and trailer-filling facility, machine shop, trailer maintenance area, and is the location of the Branch of Engineering's Section of Technical and Analytical Services Laboratory.
6. Exell Helium Plant (built in 1943) - 30 miles north of Amarillo, purifies and ships gaseous and liquid helium. Helium is removed from natural gas by a process that uses cryogenic (temperatures as low as -340 °F or -206.67 °C) or adsorption (ambient temperature) processes. Today we will demonstrate the effects of cryogenic temperatures.
7. Helium is in the gaseous phase and is separated from natural gas by the removal of water, carbon dioxide, and hydrogen. Then it is purified by lowering the temperature until most of the other components are liquid, or by pressure swing adsorption of other impurities.
8. Today's presentation will present the changes in properties of materials at cryogenic temperature using liquid nitrogen.

9. Helium

   a. Cylinder of helium.
   b. Balloon (inflate balloon before using in the demonstration to make sure it isn't defective) "Mylar" balloon versus rubber balloon (mass spectrometer test).
   c. Sintered crucible.
   d. Dewars.
   e. Core samples

10. Liquid Nitrogen (change of state)

    a. Expansion 710:1 (depending on temperature and pressure).
    b. Cryogenic thermometer, immerse in liquid N_2 in glass dewar.
    c. Erlenmeyer flask.
    d. Balloon filled by liquid N_2 in Erlenmeyer flask.
    e. Tea kettle, add liquid N_2 to kettle.
    f. N_2 corkgun, add liquid N_2 to cork gun.

11. Carbon dioxide cryopumping

    a. Cylinder of carbon dioxide.
    b. Balloon (test for holes).
    c. Test tube.
    d. Liquid N_2 in glass dewar.

12. Property changes

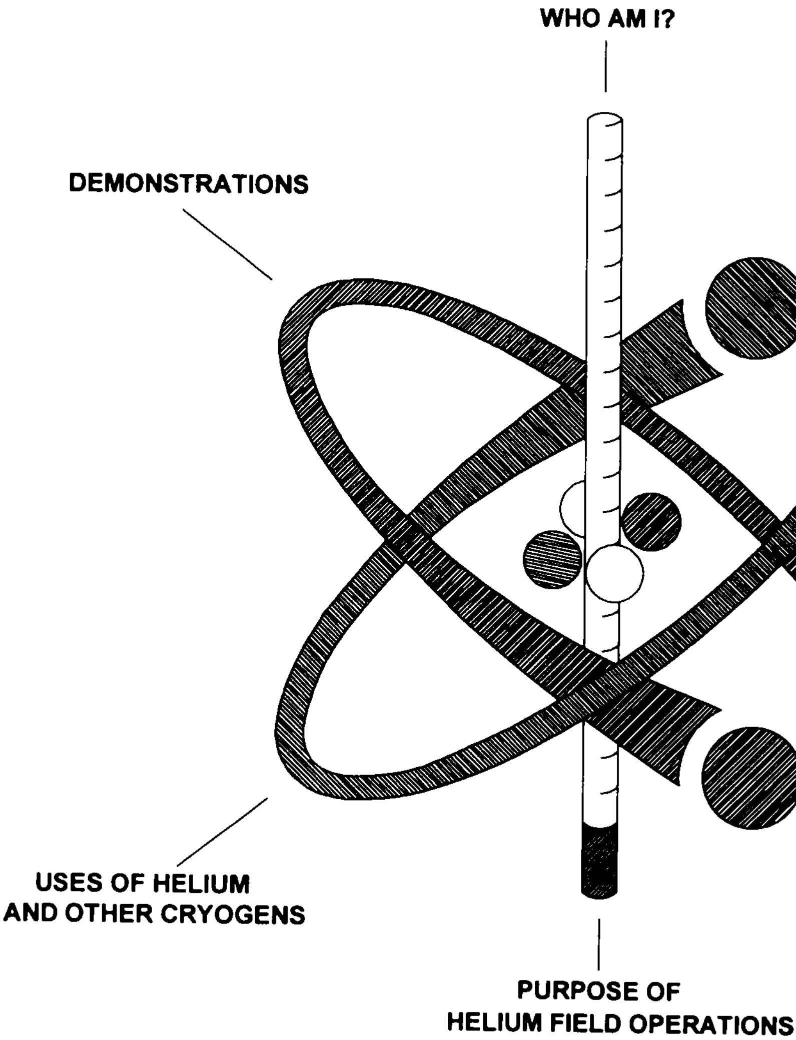
    a. Hollow ball (racquetball).
    b. Rubber tubing.
    c. Banana + nail + wood block.
    d. Flashlight with external copper coil.
    e. Lead bell.
    f. Flowers shatter when frozen.
    g. Stainless steel versus conduit steel at cryogenic temperatures.

13. Removal of heat by vaporization.

    a. Cryophorus.
    b. Liquid N_2 in glass dewar.

14. Close demonstration with question and answer session.

APPENDIX C



CRYOGENICS PRESENTATION OVERVIEW

APPENDIX D

Most Frequently Asked Questions

1. What is cryogenics?

ANSWER: The science of very low temperatures and their effect on the properties of matter.

2. How do you get the temperature that low?

ANSWER: By expanding gas through an expansion valve and heat exchangers the heat is removed and the temperature is lowered.

3. How does freezing burn?

ANSWER: The cryogenic liquids remove heat from the living tissues, freezing the H_20 and rupturing the cells.

4. How does helium change your voice?

ANSWER: The helium changes the frequency with which the voice resonates. It is not safe to breath high pressure helium (See Appendix G).

5. How much are chemists paid?

ANSWER: In 1995, Government chemists basic annual salaries were paid on the basis of the GS grade levels, (GS-5/1) $18,707 - (GS-15/10) $88,326. Salaries are adjusted by locality pay, and an extra $100,000 could be made through income from patents.

6. Can I have pieces of the broken racquetball?

ANSWER: <u>After</u> the demonstration, small pieces of the broken rubber material can be given to those who want them.

7. How do you "mine" helium?

ANSWER: Helium is "mined" by drilling for natural gas then removed before the natural gas is marketed.

8. Where does helium come from?

ANSWER: From radioactive disintegration of uranium and thorium. The helium is then trapped in underground reservoirs with natural gas. Most of the helium that is recovered commercially is located the natural gasfields of the Texas and Oklahoma Panhandles and Southwestern Kansas.

9. How is helium made?

ANSWER: From the disintegration of uranium and thorium.

10. What can you do with helium?

ANSWER: With helium you can: make lasers, shield material from air, lift balloons, guide missiles, create strong welds, make breathing mixtures, propel rocket fuel, check for leaks, make electrical parts, fabricate computer chips, build improved wind tunnels, support superconductivity, provide cryogenic pumping, and much more.

11. Why does metal break when cooled?

ANSWER: The cooling reduces the malleability and changes the crystalline structure of the metal.

12. What is superconductivity?

ANSWER: The ability of electric current to flow with zero resistance.

13. How is helium stored?

ANSWER: USBM helium is stored in an underground gasfield in the geologic strata of the Bush Dome.

14. Why don't cylinders float away?

ANSWER: The lifting capacity of a fixed volume of helium decreases since gas becomes heavier as it is compressed.

15. Since helium is lighter than air, how much does helium in a cylinder weigh?

ANSWER: The helium in a cylinder at 2,400 psig weighs 2.83 pounds or 1.28 kg.

APPENDIX E

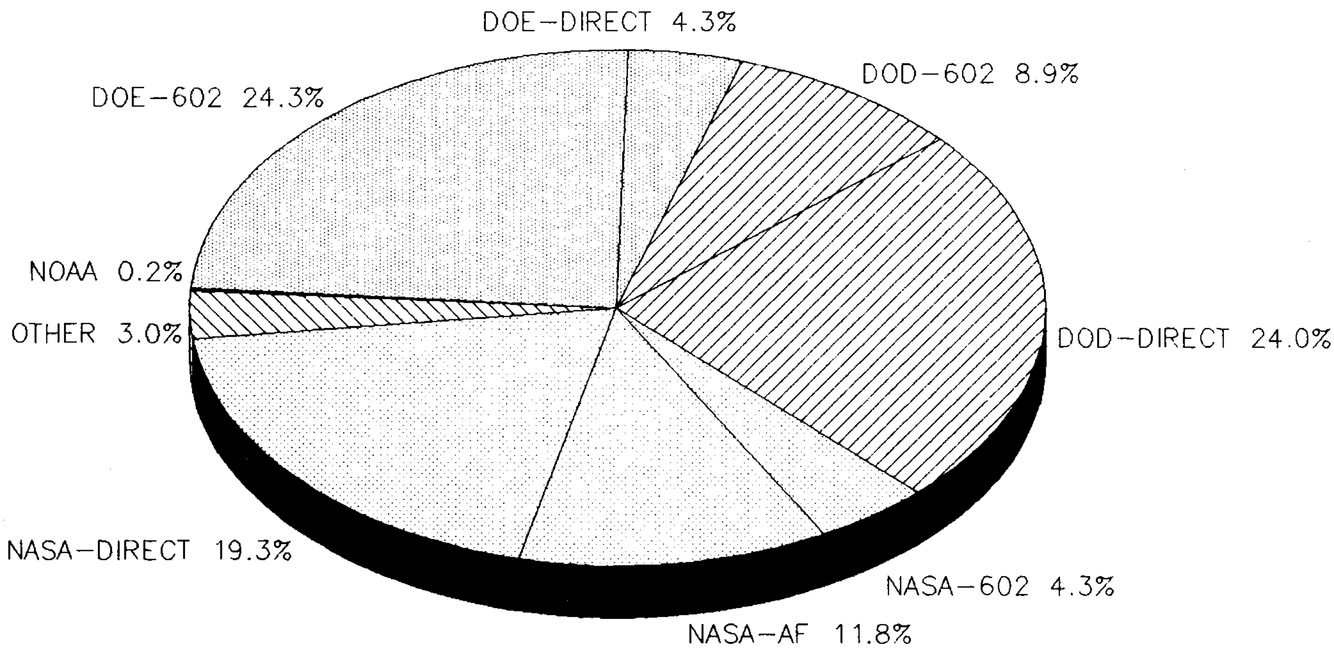
Helium Users

NASA
Departments of the Army, Navy, and Air Force (DOD)
Department of Energy (DOE)
NOAA (Weather Service)
United States Customs Service
Nuclear Regulatory Commission
University research centers
Hospitals

APPENDIX F

FEDERAL AGENCY SALES

(Total FY-91 Federal Agency sales = 349.2 MMcf)



APPENDIX G

Helium Inhalation Memorandum

December 18, 1989

Memorandum

To: Helium Field Operations Employees

From: Safety Manager

Subject: Helium Inhalation for "Donald Duck" effect

Recently the Compressed Gas Association "CGA" wrote the producers of the movie "Parenthood" about the dangers of a scene in the movie in which a grandmother inhales helium from a balloon to give her voice the squeaky Donald Duck sound. The CGA warns that "two deep breaths, one to purge the lungs of air, and the second to fill up with helium, can cause death."

Last year, three people died from inhaling helium for the voice-changing effect. One death was from using an unregulated cylinder, which exploded the victim's lungs. The other two were due to inert-gas asphyxiation, the hazard of which is that there is no warning of the danger, only collapse with no chance of self-help.

Helium is a substance foreign to the body, and like so many others can have unforeseen effects when ingested. Please alert others to the danger of breathing helium when you see others try it.

Rudy J. Wesel

INT.BU.OF MINES,PGH.,PA 30218

☆ USGPO 709-009/20,074