



AEROSPACE INFORMATION REPORT

AIR 839B

Society of Automotive Engineers, Inc.
TWO PENNSYLVANIA PLAZA, NEW YORK, N.Y. 10001

Issued 1-10-65
Revised 4-1-69

RECOMMENDED MATERIALS AND PRACTICES FOR USE WITH CRYOGENIC PROPELLANTS

FOREWORD

This report presents the second revision to "Recommended Materials and Practices for Use with Cryogenic Propellants" which was originally issued as SAE AIR 839 on 1/10/65 and revised on 4/15/67. The first revision has been completely updated, and one additional fluid - helium - has been incorporated in this latest edition.

The information presented in this document is meant to provide a basis for summary evaluation of selected cryogenic propellants. An attempt has been made to furnish a coverage which is broad in scope, yet comparatively brief in content, considering the amount of material involved. The information has been compiled from sources in the unclassified literature wherever possible; in this regard, extensive use has been made of material presented in The Handling and Storage of Liquid Propellants manual prepared for the Office of the Director of Defense Research and Engineering by a work group of the Advisory Panel on Fuels and Lubricants, the Liquid Propellant Manual prepared by the Liquid Propellant Information Agency at the Johns Hopkins University, Silver Spring, Maryland, and the Manual for Handling Missile Propellants (AFMTC TR 58-7) prepared for the Air Force Missile Test Center, Patrick AFB, Florida, by Pan American World Airways, Inc.

Inclusion of a comprehensive annotated bibliography and a tabular summary of the physical properties of each propellant - providing a ready source to the original references - allows the user to perform his own evaluation of the data if he so desires. The bibliography offers a more complete coverage of subject material than anything heretofore available in the literature; annotations should increase the value of this section. Physical property values presented in this work are "interim values" taken from selected references by the Cryogenic Data Center of the National Bureau of Standards Cryogenic Engineering Laboratory. An evaluation program is under way to determine the "most-probable" or "best" values.

Prepared by SAE Committee AGE-1, Military and Space Support Equipment (Fluid Systems Subcommittee). Particular acknowledgment is given to committee member Alan F. Schmidt for the selection, compilation, and organization of the information.

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I. RECOMMENDED MATERIALS AND PRACTICES FOR USE WITH CRYOGENIC PROPELLANTS

- A Compilation from the Literature with Annotated Bibliography -

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1. INTRODUCTION

1.1 General - Liquid propellant fuels and oxidizers are, in general, highly reactive chemicals. Consequently, the propellants and their reaction products possess certain hazardous properties which must be fully understood by all who are required to handle them. This manual is intended as a guide to help establish rules as needed to fit actual cases. The fitting of details into this framework must be left to personnel assigned to specific operations. Accordingly, information on the properties of these materials is given so that hazards will be recognized and understood.

1.2 Personnel Training and Supervision - All operating personnel shall be taught the nature of propellants and the general principles of safe conduct in handling, storage, and use of such materials. Propellants described here can be handled safely when certain simple basic principles are known and followed faithfully. On the other hand, ignorance or carelessness can result in permanent injury or death. Each person engaged in this work should be taught procedures of self-aid and first-aid, which if applied promptly afford a substantial measure of protection against injury in the event of accident.

2. HAZARDS

The three hazards to be dealt with in operations involving cryogenic propellants are health, fire, and explosion. Spills pose serious problems in inhabited areas since the low-temperature fluids and vapors may lead to physiological hazards such as frostbite and asphyxiation, and in some cases, to fires and explosions.

2.1 Health - Considering the response of the human body to its environment, appropriate engineering design and operating procedures must be exercised to minimize spills

and leaks and to insure adequate ventilation. Local exhaust ventilation is usually preferable to general ventilation because it controls contamination of air at the source. The choice between the two types of ventilation will normally be dictated by the nature of the specific operation.

Adequate water supplies must be available for instant use in an appropriate form, such as showers and eye-wash fountains. Water is the best single agent for minimizing burns by propellants.

As a general guide to the control of toxic vapors, gases, and mists, hygienic standards such as Threshold Limit Values (often referred to as MAC's or Maximum Allowable Concentrations) are useful. There are other gases, such as hydrogen, nitrogen, and helium, which though not toxic in the usual sense, can cause asphyxiation by exclusion of oxygen from the immediate environment.

2.2 Fire - Each working area must be kept free of combustible materials. Ventilation is essential for keeping vapor and dust concentrations at a safe level. Fuels and oxidizers must be kept separated; fire-fighting equipment and extinguishing agents must be suited to the type of fire which may occur with a particular fuel or oxidizer. Where flammable vapors may be present, electrical equipment must be of the approved explosion-proof type and equipment and containers grounded. Nonstatic general wearing apparel should be provided and used where necessary. Approved lightning protection systems should be installed where required.

2.3 Explosion - Principles guiding the elimination and control of fire hazards are generally applicable to explosion hazards as well. In addition, earth, sand, concrete, or metal barricades of sufficient mass should be provided in appropriate places to lessen the effects of an explosion. Shock-sensitive materials must be protected from vibration and impact.

3. SAFETY MEASURES

Properly engineered systems are paramount to safe usage of propellants. In the construction of such systems, only materials compatible with the particular propellants for which the system is designed can be used. In order to eliminate the possibility of an accident, the entire system must be cleaned properly to eliminate any foreign matter.

Successful control of hazards requires thorough training of personnel in the following subjects:

1. The chemical and physical characteristics of the fuels and oxidizers.
2. The hazards peculiar to each fuel and oxidizer and the proper personal protective measures required.
3. The most efficient technique required for control of fires involving a specific fuel or oxidizer.

4. TRANSFER AND STORAGE

Fire and explosion hazards have an important influence on the design of main storage units and on their location with respect to each other and to populated buildings and areas. Fuels and oxidizers must be separated by distance or barriers, and ready storage quantities should consist only of the minimum amount necessary for the operation. Storage tanks should be diked, and ventilation should take advantage of prevailing winds and natural terrain.

Ample water for fire-fighting and decontamination must be provided. Cleanliness must be observed throughout the area, and rules regarding safe practices must be rigidly enforced.

All operations with cryogenic fluids should be planned carefully, and a set of preparatory, operating, and emergency procedures should be prepared for the operating personnel; these procedures should cover special cleaning and fire-fighting instructions, required clothing, safety devices, etc.

GENERAL BIBLIOGRAPHY

A general listing by title and mode of publication is presented below, followed by technical literature abstracts of each item:

Books

Cryogenic Engineering
Cryogenic Technology
Applied Cryogenic Engineering
Cryogenic Systems
Cryogenics
Safety with Cryogenic Fluids
Rocket Propellant and Pressurization Systems
Ground Support Systems for Missiles and Space Vehicles

Conference Proceedings

Advances in Cryogenic Engineering - Proceedings of the National Cryogenic Engineering Conferences
Cryogenic Safety - A Summary Report of the Cryogenic Safety Conference
Proceedings of the Propellant Thermodynamics and Handling Conference
Proceedings of the Conference on Long-Term Cryo-Propellant Storage in Space

Manuals

The Handling and Storage of Liquid Propellants
Liquid Propellant Manual
Manual for Handling Missile Propellants
General Safety Precautions for Missile Liquid Propellants
Liquid Propellants Safety Handbook
Handling Hazardous Materials

Compendiums, Handbooks, Monographs, Surveys

A Compendium of the Properties of Materials at Low Temperatures (Phase I)

A Compendium of the Properties of Materials at Low Temperatures (Phase II)
Cryogenic Materials Data Handbook
Mechanical Properties of Structural Materials at Low Temperatures
Properties of Materials at Low Temperatures
Specific Heats and Enthalpies of Technical Solids at Low Temperatures
Thermal Expansion of Technical Solids at Low Temperatures
Infrared Reflectances of Metals at Cryogenic Temperatures
Tensile and Impact Properties of Selected Materials from 20 to 300°K
Cryogenic Data Book
Properties of Selected Rocket Propellants
Compatibility of Materials with Rocket Propellants and Oxidizers
Compatibility of Plastics with Liquid Propellants, Fuels and Oxidizers
Properties of Plastics and Related Materials at Cryogenic Temperatures
Metals and Alloys for Cryogenic Application - A Review
Effects of Low Temperature on Structural Materials
Low Temperature and Cryogenic Steels
Aerospace Fluid Component Designers Handbook
Advanced Valve Technology
Cryogenic Valves . . . a Survey
Cryogenic Control Valves for Industrial Processes
A Broad Survey of Cryogenic Pumping
Handbook of Compressed Gases
Handbook of Thermal Design Data for Multilayer Insulation Systems
Thermal Insulation Systems
Boiling Heat Transfer for O₂, N₂, H₂, and He

Boiling Heat Transfer for Cryogenics
 Review of Static Seals for Cryogenic Systems
 Bearings and Seals for Cryogenic Fluids

Cryogenic Information Report
 Cryogenic Technology - Journal of the Cryogenic
 Society of America

Miscellaneous Publications, Papers, Reports

Introduction to Cryogenics
 Introduction to Cryogenic Engineering
 The Storage and Handling of Cryogenic Liquids
 Safe Handling of Cryogenic Fluids
 Air-Condensing Cryogenic Fluids
 Air-Solidifying Cryogenic Fluids
 Cryogenic Safety
 Fire-Hazard Properties of Flammable Liquids,
 Gases and Volatile Solids
 Review of Fire and Explosion Hazards of Flight
 Vehicle Combustibles
 Explosion and Fire Hazards Associated with the Use
 of Low-Temperature Industrial Fluids
 Sparking Characteristics and Safety Hazards of
 Metallic Materials
 Problems in Cryogenics
 Thermal Problems Peculiar to Cryogens in Space
 Cryogenic Instrumentation
 Cryogenic Thermocouple Thermometry
 Mass Flowmeters in Cryogenic Service
 Cryogenic Flow Measurement - Flow Meters
 Flow Measurement of Cryogenic Fluids
 Large Size Cryogenic Turbine Type Flowmeter
 Technology
 Cooldown of Large-Diameter Liquid Hydrogen and
 Liquid Oxygen Lines
 Cooldown of Cryogenic Transfer Systems
 Flexibility Considerations for the Design of
 Cryogenic Transfer Lines
 Cryogenic Piping System Design Considerations
 Fundamentals of Low Temperature Piping Design
 Cryogenic Storage Vessels
 How to Specify Low Temperature Storage Vessels
 Design of Cryogenic Storage Tanks for Industrial
 Applications
 Large Cryogenic Storage Vessels
 Construction of High Pressure Storage Vessels
 The Past, Present and Future of Metals for Liquid
 Rockets
 Low Temperature and Cryogenic Steels: How to
 Choose, Use, and Weld Them

Bibliographies

Rocket Propellants, DDC (AD 233 500)
 Rocket Propellants, DDC (AD 315 500)
 Rocket Propellants, DDC (AD 263 000)
 Rocket Propellants, DDC (AD 325 000)
 Cryogenics and Low Temperature Research,
 DDC (AD 271 000)

Journals

Cryogenics - An International Journal of Low
 Temperature Engineering and Research
 Cryogenic Engineering News

CRYOGENIC ENGINEERING - Scott, R. B. - D. Van
 Nostrand Company, Inc., Princeton, New Jersey (1959)
 368 pp.

Throughout the preparation of this book an objective al-
 ways considered was to present the necessary information in
 such a manner that an investigator with a new idea involv-
 ing cryogenic techniques can assess the feasibility of his pro-
 ject and gain some idea about the difficulties that he should
 expect. Because this book is intended primarily for the
 reader who is unfamiliar with low temperatures, the treat-
 ment is deliberately elementary, but it is not trivial. It is
 believed that considerations of practical importance can be
 presented in language that is easily understood. There is
 little or no attempt to deal with either the esoteric concepts
 of modern cryogenic physics or the refinements that engi-
 neering practice has established in some disciplines used by
 cryogenists such as heat exchange, distillation, or adsorp-
 tion. References to authoritative information on such sub-
 jects are given. The emphasis here is upon both basic and
 applied information most important in engineering research
 and development at low temperatures.

Since this treatise emphasizes the practical aspects of
 low-temperature technology, it is hoped that the informa-
 tion will be most useful to the design engineer who has the
 responsibility of making "practical" equipment work.

CRYOGENIC TECHNOLOGY - Vance, R. W. - John
 Wiley & Sons, Inc., New York (1963) 585 pp.

The goal of this treatise is to provide a source or refer-
 ence for those engaged either in applications of, or basic,
 theoretical studies. The text material contains an analysis
 of thermodynamic principles and cycles describing in de-
 tail how low temperatures are achieved, followed by dis-
 cussions of the properties of liquids and solids, applications
 of phase equilibria relationships to industrial processes, heat
 transfer, thermometry, insulation techniques, and the fun-
 damental theories involved in superconductivity. To aug-
 ment this theoretical background are discussions of some of
 the principle applications of cryogenic engineering such as
 the cryotron and other superconductive devices, the solid-
 state maser, space simulation and cryopumping, nuclear
 propulsion, the safety aspects and explosive potentials of
 cryogenic propellants, and deep-space probes, including
 the cryogenic storage problems in extraterrestrial environ-
 ments. An analysis of cryobiology technology provides an
 unusual climax.

APPLIED CRYOGENIC ENGINEERING - Vance, R. W. and
 Duke, W. M. - John Wiley & Sons, Inc., New York (1962)
 510 pp.

This compilation of lectures is designed to enhance the
 knowledge of scientists and engineers engaged in research
 and development as well as operating personnel, in the field
 of cryogenics. It is particularly applicable to missile and
 space vehicle systems, but the basic theory explained is ap-
 propriate to all fields of endeavor requiring technical skills

in the application of low temperatures to industrial processes.

The book has been divided into two parts: Part I - Basic Theory; Part II - Applications of Cryogenic Engineering.

Part I discusses basic theory, beginning with the properties of cryogenic fluids and covering mechanical properties of materials in cryogenic environments, low temperature thermometry, fluid flow and heat transfer, insulation techniques and some non-missile applications of cryogenic equipment. It consists of eight chapters in which the theories applicable to the use of cryogenic materials in ballistic missiles and space vehicles are condensed into engineering form augmented by organized experimental data.

Part II describes many of the applications of cryogenic theories to space vehicles and systems. Also, predictions have been made as to what the future propulsion systems might be for space vehicles.

CRYOGENIC SYSTEMS - Barron, R. - McGraw Hill Company, Inc., New York (1966) 678 pp.

The objective of this book is to present an introduction to the engineering aspects and challenges of cryogenics. Emphasis is placed on the design and analysis of systems used to produce, maintain, and utilize low temperatures. The text is an outgrowth of class notes and lecture material associated with a course in cryogenic systems taught at Ohio State University and is slanted primarily toward senior mechanical engineering students, although the text is arranged so that it may be used by an engineer unfamiliar with cryogenic techniques when he is called upon to assist in the design of a system for low temperatures.

CRYOGENICS - McClintock, M. - Reinhold Publishing Corp., New York (1964) 270 pp.

The purpose of this book is to give to the engineer or scientist with another specialty, or to the educated non-scientist, a qualitative understanding of the basic aspects and some of the representative applications of cryogenics.

SAFETY WITH CRYOGENIC FLUIDS - Zabetakis, M. G. - Plenum Press, New York (1967) 156 pp.

This monograph provides a concise, complete exposition of the principles of safety in the handling of cryogenic liquids. It covers safety rules, design data, first aid precautions, and hazard control procedures while stressing the basic principles of handling low-temperature materials and the properties of cryogenic fluids. Thus it permits the reader to design his own unique operation with assurance and security even when the physical situation is not specifically covered by standard safety engineering rules.

ROCKET PROPELLANT AND PRESSURIZATION SYSTEMS - Ring, Elliot (Editor) - Prentice-Hall, Inc., Englewood Cliffs, N. J. (1964) 310 pp.

Presented here are those subjects which make up the entire propulsion field, engine, propellant, feed systems, and pressurization systems, and the specific problems in each. Included are sections on cryogenic loading problems, geysering, stratification, special zero gravity fluid

problems, propellant hardware, instrumentation, etc. The book is designed to deal in depth with such problems.

GROUND SUPPORT SYSTEMS FOR MISSILES AND SPACE VEHICLES - Brown, K. and Weiser, P. - McGraw-Hill Company, Inc., New York (1961) 490 pp.

The aim of this book is to present a complete picture of the systems required to support either a missile or a space vehicle. Liquid propellant handling considerations are covered in Part IV (book is divided into five parts) and include a detailed discussion on cryogenics, hazards and safety, and the design and development of fast-fill propellant loading systems.

ADVANCES IN CRYOGENIC ENGINEERING - Timmerhaus, K. D. (Editor) - Advances in Cryogenic Engineering Vols. 1-13, Plenum Press, New York (1960-68)

A series of volumes dealing with low temperature phenomena and their applications; each volume constitutes the Proceedings of one Cryogenic Engineering Conference, beginning with Volume I - Proceedings of the First National Cryogenic Engineering Conference held at the National Bureau of Standards, Boulder, Colorado (September 1954) and covering all subsequent conferences to date.

CRYOGENIC SAFETY - A SUMMARY REPORT OF THE CRYOGENIC SAFETY CONFERENCE - Air Products, Inc., Allentown, Pa., (July 1959) 145 pp 77 fig 12 tab 61 ref.

This report covers material presented in five sessions and eleven seminars at the 1959 Cryogenic Safety Conference. The session topics included (1) Hazards of Cryogenic Systems, (2) Technical Character of Cryogenic Hazards, (3) Design and Construction for Safety, (4) Operations for Safety, and (5) Liquid Hydrogen Safety.

PROCEEDINGS OF THE PROPELLANT THERMODYNAMICS AND HANDLING CONFERENCE - Bollinger, L. E. and Lemmons, A. W. (Editors) Proc. of Propellant Thermodynamics and Handling Conference, Ohio State Univ. (July 20-21, 1959) O. S. U. Eng. Experiment Station Rept. No. 12.

The "Propellant Thermodynamics and Handling Conference" was one of the first "specialist" conferences held by the American Rocket Society. Coverage of two technical areas was planned originally for the conference: propellant thermodynamics and propellant handling. The propellant handling area had papers covering such aspects as materials problems, handling techniques, safety, and toxicity of high-energy and cryogenic liquid propellants and their combustion products.

Included in this collection are the following titles:

The Large Scale Production, Handling, and Storage of Liquid Hydrogen.

Safety Aspects in the Handling and Storage of Liquid Hydrogen.

Experience with Handling Liquid Hydrogen in Engine Testing.

Transportation, Transfer and Storage of Liquid Fluorine. Materials of Construction for Handling Fluorine.

**Some Problems in Using Fluorine in Rocket Systems.
Safety and Handling of Ozone-Oxygen Mixtures.**

PROCEEDINGS OF THE CONFERENCE ON LONG-TERM CRYOPROPELLANT STORAGE IN SPACE - NASA - Marshall Space Flight Center, Huntsville, Ala. (Oct. 12-13, 1966) 264 pp.

This conference was planned to promote long-term storage of cryogenic propellants in space. The papers represent a cross section of significant topics on design, testing and instrumentation. Included in this collection are the following titles:

Thermal Mass Penalties Associated with Cryogenic Propellant Storage on Manned Mars and Venus Missions.
Lunar Storage of Cryogenic Reactants.

Development of Materials and Materials Application Concepts for Joint Use as Cryogenic Insulation and Micrometeoroid Bumpers.

Thermal Problems Associated with the Development of a Flight Configured Cryogenic Insulation System.
Shadow Shield Experimental Studies.

Performance of a LH₂ Shrouded Supercritical Helium System.

Study of Vapor/Liquid Separators for Venting Cryogenic Propellant Tanks in Space.

Characteristics of Two-Phase Flow in a Choked Nozzle.
Thermodynamic Model for Orbital Cryogenic Propellant Tanks with P-T Correlations for Saturn S-IV Stage and Centaur Flights.

Topics Requiring Further Study on the Effect of Gravity upon Heat Transfer.

The Growth of Hydrogen Bubbles in Low Gravity.
Finite Difference Solution of Stratification and Pressure Rise in Containers.

Flow Visualization and Thermal Stratification of Water in a Horizontal Cylinder and a Sphere.

Vapor Pull-Through at a Tank Drain with and without Dielectrophoretic Baffling.

Gradient Stabilization of Electrohydrodynamically Oriented Liquids.

Heat Transfer to Rotating Cryogenic Fuel Tanks in Orbit.

Low Gravity Fluid Behavior and Heat Transfer Results from the S-IV B-203 Flight.

Liquid-Solid Hydrogen Mixtures as Vehicle Propellants.
Slush Hydrogen Characteristics.

Cryogenic Transparent Tank Development.

THE HANDLING AND STORAGE OF LIQUID PROPELLANTS - Chemical Propulsion Information Agency, Defense Research and Engineering, Wash., D. C. (Jan. 1963) 338 pp.

This manual is published as a source of information; it is intended for use as a basis for the preparation of regulations governing the handling and storage of liquid propellants.

Included in the contents are four chapters on cryogenic propellants (fluorine, oxygen, nitrogen, hydrogen), fifteen chapters on non-cryogenic propellants, and appendices covering fire fighting techniques, fire protection and extinguishment, cleaning procedures and hazard characteristics.

LIQUID PROPELLANT MANUAL - Liquid Propellant Info. Agency, Johns Hopkins Univ., Silver Springs, Md., Manual (March 1961) Contr. No. NOrd 7386.

The liquid propellant manual is a compendium of the properties of liquid propellants, and is intended to provide a survey of the physico-chemical properties of selected propellants and promising propellant candidates in order to assess their performance or feasibility in rocket applications. Additional information and revisions will be issued at frequencies depending upon the receipt of new data. Units for specific elements, compounds, and mixtures used as liquid propellants will contain information on the physical, chemical, thermodynamic, transport, and electromagnetic properties for these materials. In addition, information on safety, handling, compatibility of materials, and logistics will be included.

MANUAL FOR HANDLING MISSILE PROPELLANTS - Pan Am. World Airways, Patrick AFB, Fla., AFMTC Rept. No. TR 58-7 (1958) DDC AD 184 277.

This manual presents in a single authority the miscellaneous information available from diverse sources. It combines such data with practical experience and offers a standard of operation as current as possible. It deals with the hazards involved in the storage, handling, and the use of propellants employed in rocketry. The procedures and cautions recommended herein should be adhered to for the safety of personnel and property. It is intended as a guide and an outline of the most important precautions to be taken in operations involving certain hazardous fuels, oxidizers, and other chemicals. This publication is not to be considered as an engineering manual or as a design handbook; neither should it restrict development of new methods and procedures. It can be called a list of "things to do" and "not to do" for maintaining a reasonable degree of safety relating to the materials discussed. These procedures, in combination with the general principles of industrial safety, intelligently utilized should result in accident-free operations.

LIQUID PROPELLANTS SAFETY HANDBOOK - NASA-Kennedy Space Center (Safety Office), Cocoa Beach, Fla. - NASA Rept. SP-4-44-S (April 1, 1965).

This handbook contains the latest information concerning liquid propellants utilized by the NASA at the Kennedy Space Center at Merritt Island Launch Area as of the date of this publication. Changes will be issued periodically to keep the handbook current and accurate.

HANDLING HAZARDOUS MATERIALS - NASA - Technology Utilization Division - NASA Rept. SP-5032 (Sept. 1965), 93 pp.

This publication deals with highly reactive materials that have been studied in the search for fuels and oxidizers for space work: Liquid hydrogen, fluorine, ozone (and several non-cryogenics). It describes both the hazards that have restricted the use of these materials and the procedures by which they have been handled and stored safely. References are given to work done by NASA and other investigators.

GENERAL SAFETY PRECAUTIONS FOR MISSILE LIQUID PROPELLANTS - Air Force Tech. Order 11C-1-6 (Oct. 18, 1961).

The purpose of this technical order is to provide safety measures, safety standards, procedures, instructions, and precautions for preventing fires, explosions, personal injuries, poisonous and injurious conditions, and property damage pertaining to:

- Servicing operations at test facilities and launch complexes involving missile liquid propellants.
- The construction, operation, or alteration of test facilities and launch complexes designed for the testing, servicing, or launching of liquid propulsion systems or missiles.
- The arrangement or safeguarding of equipment, machinery, plumbing, tanks, containers, and accessories for operating buildings and technical facilities.

This publication is not to be considered as a design manual or engineering handbook on missile liquid propellants, nor is it intended to restrict development in this field. Its primary purpose is to present the things "to do" and "not to do" to obtain reasonable and adequate safety in the operational handling and use of highly reactive chemicals.

A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURES - PHASE I: Part I. Properties of Fluids (July 1960); Part II. Properties of Solids (Oct. 1960); Part III. Bibliography of References (Oct. 1960). Johnson, V. J. (General Ed.) Natl. Bur. Standards Cryogenic Eng. Lab., WADD Tech. Rept. 60-56, Contr. No. AF 33(616)58-4.

Phase I of the Compendium is divided into three parts: Part I, ten properties of ten fluids; Part II, three properties of solids; Part III, an extensive bibliography of references. Density, expansivity, thermal conductivity, specific heat and enthalpy, transition heats, phase equilibria, dielectric constants, adsorption, surface tension and viscosity for the solid, liquid and gas phases of helium, hydrogen, neon, nitrogen, oxygen, air, carbon monoxide, fluorine, argon and methane are given wherever adequate data could be collected. Thermal expansion, thermal conductivity and specific heat and enthalpy are given for a number of solids of interest in cryogenic engineering. Data sheets, primarily in graphic form, are presented from "best values" of data collected. The source of the material used, other references and tables of selected values with appropriate comments are furnished with each data sheet to document the data presented. Conversion tables and other helpful information are also included.

A COMPENDIUM OF THE PROPERTIES OF MATERIALS AT LOW TEMPERATURE - PHASE II, Part IV. Stewart, R. B. and Johnson, V. J. (General Ed.) Natl. Bur. Standards Cryogenic Eng. Lab. WADD Tech. Rept. 60-56 (1961) Contr. No. AF 33(616)59-6, 501 pp.

Phase II of the Compendium includes data sheets on compressibility factor, velocity of sound and entropy of fluids, vapor-liquid equilibrium concentration of binary mixtures of fluids, and electrical resistivity and thermal conductivity

integrals of metallic solids. Data sheets are included for each of these properties for the following materials: Compressibility Factor (Helium, Hydrogen, Neon, Nitrogen, Air, Methane); Entropy (a T-S diagram for Neon); Velocity of Sound (in liquids: Helium, Hydrogen, Nitrogen, Oxygen, Argon, Methane; in gases: Helium, Hydrogen, Neon, Nitrogen, Oxygen, Air, Carbon Monoxide, Methane; Nitrogen in Oxygen, Carbon Monoxide, Argon, Methane); Electrical Resistivity (53 of the pure metallic elements); and Thermal Conductivity Integrals (44 pure metallic substances, 36 non-ferrous alloys, 9 ferrous alloys and 4 glasses and plastics). In general the data sheets present the data primarily in graphical form, and in addition include tables of selected values, references to the sources of the data and other references. Appropriate comments of interest to the user are also given.

CRYOGENIC MATERIALS DATA HANDBOOK - Durham, T. F., McClintock, R. M. and Reed, R. P. - Natl. Bur. Standards Cryogenic Eng. Lab. Contr. No. AF 04(647)-59-3, 556 pp 1107 ref.

This handbook of data on solid materials at low temperatures contains certain mechanical and physical properties of selected metals and non-metals over the temperature range minus 454°F to plus 500°F. The materials are mostly ones in current use for missile applications at cryogenic temperatures, but a few have been included because of their potential for such uses. The properties are those which are most generally useful to the designer. The compilation is believed to include all reliable data which have appeared in the literature from 1940 thru 1959 and recent data from test laboratories. In some cases the data reported have not yet been published. Information from a few papers published prior to 1940 has also been included.

MECHANICAL PROPERTIES OF STRUCTURAL MATERIALS AT LOW TEMPERATURES; A COMPILATION FROM THE LITERATURE - McClintock, R. M. and Gibbons, H. P. Natl. Bur. Standards Monograph 13 (June 1960) 180 pp 104 ref.

The tensile strength, yield strength, tensile elongation, and impact energy of about two hundred materials, metallic and non-metallic, are given graphically as functions of temperature between 4° and 300° Kelvin.

PROPERTIES OF MATERIALS AT LOW TEMPERATURES - Corruccini, R. J. - Chem. Eng. Progress 53, 262-67, 342-46, 397-402 (A three-part article) (1957).

This review summarizes the knowledge acquired from work at the (NBS) Boulder Laboratory on low-temperature data, organized and interpreted in the light of theory, and shows how useful estimation procedures may sometimes be derived from theory to fill gaps in the available data.

SPECIFIC HEATS AND ENTHALPIES OF TECHNICAL SOLIDS AT LOW TEMPERATURES; A COMPILATION FROM THE LITERATURE - Corruccini, R. J. and Gniewek, J. J. - Natl. Bur. Standards Monograph 21 (Oct. 1960) 20 pp 4 tab 165 ref.

Tables are given of the specific heat, cp, and the enthalpy of 28 metals, 3 alloys, 8 other inorganic substances, and 8 organic substances in the temperature range, 1° to 300°K.

THERMAL EXPANSION OF TECHNICAL SOLIDS AT LOW TEMPERATURES; A COMPILATION FROM THE LITERATURE - Corruccini, R. J. and Gniewek, J. J. - Natl. Bur. Standards Monograph No. 29 (May 1961) 22 pp 4 tab 246 ref.

Tables are given of the linear contraction relative to 293°K, $(L_{293} - L_T)/L_{293}$, and the linear expansion coefficient, dL/L_{293dT} , of thirty elements, forty-five alloys, twenty-two other inorganic substances and twenty plastics and elastomers in the temperature range, 0 to 300°K.

INFRARED REFLECTANCES OF METALS AT CRYOGENIC TEMPERATURES - A COMPILATION FROM THE LITERATURE - Dickson, P. F. and Jones, M. C. - Natl. Bur. Standards Tech. Note 348 (Oct. 1966) 66 pp. 11 fig. 20 tab. 33 ref.

Spectral and total reflectances for metals at cryogenic temperatures in the infrared wavelength region are compiled from the literature. Information concerning sample preparation and purity, radiation source, and methods of reflectance measurement are also presented. Observations regarding the effects on reflectance of temperature, oxide layer, wavelength, and sample preparation are given.

TENSILE AND IMPACT PROPERTIES OF SELECTED MATERIALS FROM 20 TO 300°K - Warren, K. A. and Reed, R. P. - Natl. Bur. Standards Monograph 63 (June 1963) - 51 pp 30 fig 7 tab 3 ref.

The tensile and impact properties of structural materials were experimentally determined at temperatures from 20 to 300°K. Tensile properties of a few materials were also determined at 4°K. The materials included forty-two commercial alloys of iron, aluminum, titanium, copper, nickel, and cobalt, and two metal-bonded carbides. The properties experimentally determined were the yield strength, tensile strength, elongation, and reduction of area, the stress versus strain curve, and the impact energy. The test equipment and procedures are described. The individual data are presented in tables, and the average results are displayed in graphs.

CRYOGENIC DATA BOOK (CGS UNITS) - Chelton, D. B. and Mann, D. B. (NBS-CEL, Boulder, Colo.) Calif. Univ., Radiation Lab., Berkeley, Rept. No. UCRL-3421 (May 1956) 116 pp.

Increased activities in Cryogenic Engineering have brought about the need for a compilation of available data. The purpose of the Cryogenic Data Book is to provide a condensed source of reliable data and reference information for those working in the cryogenic field. Specifically the data were compiled with a view toward the design of liquid hydrogen bubble chambers.

The compilation does not constitute a critical survey of the literature.

PROPERTIES OF SELECTED ROCKET PROPELLANTS - Vol. I (1963) Vol. II (1964) - The Boeing Company, Seattle, Washington - Document D2-11677 DDC AD 444 642.

The purpose of this document is to provide information on chemical, physical and thermodynamic properties, handling and safety characteristics, methods of production, and availability and cost of selected rocket propellants. (Cryogenic propellants hydrogen, fluorine, and oxygen are included in this work.)

COMPATIBILITY OF MATERIALS WITH ROCKET PROPELLANTS AND OXIDIZERS - Boyd, W. K., Berry, W. E., and White, E. L. - Battelle Mem. Inst., Defense Metals Inform. Center, Columbus, Ohio DMIC Memo. No. 201 (Jan. 1965) 41 pp 36 tab 302 ref.

This report summarizes the available information on the compatibility of liquid rocket propellants with prominent materials of construction. Fuels and oxidizers of current interest are discussed. The corrosion data which are presented will apply to storing, handling and control equipment outside of missiles and to missile components excluding combustion chamber. The compatibility of materials with reaction products in combustion chambers, nozzles, etc., has not been considered.

The report is subdivided into sections according to the propellant. Each material of construction is rated for a given medium as belonging to one of four classes, based primarily upon corrosion resistance. Consideration also is given to such factors as catalytic decomposition and sensitivity to impact.

COMPATIBILITY OF PLASTICS WITH LIQUID PROPELLANTS, FUELS AND OXIDIZERS - Beach, Norman E., Plastics Technical Evaluation Center, Picatinny Arsenal, Dover, New Jersey, PLASTEC REPORT 25 (Jan. 1966) 119 pp. 43 ref. - DDC AD 632 287.

Much has been published on the subject of the compatibility of plastics with liquid propellants, fuels and oxidizers, but invariably from the standpoint of the propellant or fuel. This report is a rearrangement of the published compatibility data from the standpoint of the plastic material. It is in the form of a tabulation, with primary arrangement by plastic (or elastomeric) material; and thereunder, by fuel. All arrangements are alphabetical, in the form given in the original reference; that is, either by generic or trade designation. The compatibility evaluation is in terms of the original document, briefly culled to show behavior of the material at a given temperature and for a given time. Elastomers are included (although they are not a stated concern of PLASTEC); but oils, lubricants and greases are omitted, even though based on polymers. The information has been drawn from 43 references, which are annotated so that the information extracted from them shall have additional significance.

PROPERTIES OF PLASTICS AND RELATED MATERIALS AT CRYOGENIC TEMPERATURES - Plastics Technical Evaluation Center, Picatinny Arsenal, Dover, New Jersey, Distributed under license from the IHS Archive

PLASTEC Rept. 20 (July 1965) 253 pp 46 fig 34 tab 319 ref.

This report reviews the effects of cryogenic temperatures on plastics and such related materials as elastomers and adhesives. It presents an annotated bibliography of 319 references from open literature, government project and contract reports, and conference papers. A detailed subject index and a number of supplemental indexes are included. Topics covered are: Molded Polymeric Materials (Plastics); Cryogenic Insulation; Structural Plastic Laminates; Elastomers, Seals and Sealants; Adhesives; Plastic Films, Film Laminations and Vapor Barriers; Fibers; Electrical Applications; Wear and Friction; Liquid Oxygen (LOX) Compatibility; Radiation and Combined Effects; and Miscellaneous Applications. Test Methods are not treated in a separate section in the discussion, but the subject index refers to many references with information on test procedures and apparatus.

METALS AND ALLOYS FOR CRYOGENIC APPLICATION - A REVIEW - Kendall, E. G. - Aerospace Corp., El Segundo, Calif., SSD-63-371 Rept. No. TDR-269 (4240-10)-6 (Jan. 1964) Contr. No. AF 04(695)-269, 61 pp 19 fig 11 tab 64 ref.

An up-to-date review of metals and alloys suitable for cryogenic aerospace structural applications has been made. The mechanical properties of austenitic stainless steels, other steels, aluminum alloys, titanium alloys, nickel alloys and cobalt alloys from +78 to -423°F are presented, including tensile and yield strengths, elongation and notch/tensile ratios. Mechanical properties of weldments are also presented. The question of notch toughness and the notch acuity factor, K_t , is discussed with respect to low temperature tensile testing. Compatibility with the liquid gases is discussed and alloys most suitable for containing liquid oxygen and hydrogen in aerospace vehicles are recommended.

EFFECTS OF LOW TEMPERATURES ON STRUCTURAL MATERIALS - NASA Tech. Utiliz. Rept., NASA SP-5012 (Dec. 1964) 55 pp 45 fig 37 tab 2 ref.

There are many problems, associated with the storage and handling of cryogenic fluids, which must be considered in the design and fabrication of tankage and other components of cryogenic rocket systems. Thus, a continuing program is being conducted by the Propulsion and Vehicle Engineering Laboratory of the Marshall Space Flight Center to evaluate the applicability of various metallic materials at cryogenic temperatures.

LOW TEMPERATURE AND CRYOGENIC STEELS MATERIALS MANUAL - United States Steel, Pittsburgh, Pa. - 191 pp 81 fig 30 tab 218 ref.

This manual is designed to provide knowledge of steels produced for use in low-temperature and cryogenic service. It embodies a synopsis of material properties and fabrication techniques, as well as an economic evaluation of containers and other criteria required when designing cryogenic process equipment.

AEROSPACE FLUID COMPONENT DESIGNERS' HANDBOOK - TRW Systems Group, Redondo Beach, Calif., - AFRPL TDR 64-25 (March 1967) - Contr. Nos. AF04(611) - 8385, AF04(611) - 11316, Two volumes: DDC AD 809 182, DDC AD 809 183.

The Aerospace Fluid Component Designers' Handbook is a compilation of basic information on the design, analysis, selection, and specification of valves and associated fluid components used in aerospace fluid systems. The handbook is intended to be used as a basic reference for engineers and other technical personnel who are involved in any phase of aerospace fluid component technology. It contains sections dealing with heat transfer, fluid mechanics, fluid systems, fluid components, modules, analysis, computers, specifications, contamination and cleaning, reliability, materials, and environments. The handbook will continue to be revised periodically for the purposes of maintaining the currency of the data and adding applicable new material.

ADVANCED VALVE TECHNOLOGY - Burmeister, L. C., Loser, J. B. and Sneegas, E. C. - Midwest Research Inst. - NASA SP-5019 (1967) - 183 pp 118 fig 30 tab 159 ref.

The exploration of space has necessitated a multitude of innovations and improvements in valves, the controlling elements of both simple and complex fluid-handling systems. In view of the benefits that can be derived from what has been learned about the design, performance, and manufacture of these devices, this survey was undertaken.

CRYOGENIC VALVES . . . A SURVEY - Beard, C. S. - Cryogenic Eng. News 2, 62-64; 66, 68, 69 (Nov. 1967).

The differences between cryogenic and ordinary valves are both visible and invisible. The author reviews requirements for valve bodies, materials, seats, and lubricants for compatibility with cryogens and ultra-low temperatures.

CRYOGENIC CONTROL VALVES FOR INDUSTRIAL PROCESSES - Ives, R. P. - Cryogenic Eng. News 3, 38-41 (Aug. 1968) 7 fig.

There is nothing mystic about cryogenic control valves. Their design, application and installation is simply an extension of basic control valve engineering and technology. A process or instrument engineer must review the service conditions, review the available equipment, select the unit best suited for the specific job, and apply the control valve as outlined. This does not differ from the technique used to apply a control valve to any process.

A BROAD SURVEY OF CRYOGENIC PUMPING - Hoelscher, F. W. - Cryogenic Eng. News 2, 78-80, 82, 84, 85 (Aug. 1967).

A wide-ranging outline of cryogenic pumping equipment is presented, including designs, uses, requirements, problems and limitations.

HANDBOOK OF COMPRESSED GASES - Compressed Gas Association - Reinhold Publishing Corporation, New York (1966) 416 pp.

Prepared by the staff of the Compressed Gas Association, the foremost research leader on safety and technical standards in this field, the book covers the essential core of knowledge and practice in all safe and efficient handling, storage and transportation of gases. Some of the many compressed gases included in the handbook are: acetylene, ammonia, carbon dioxide, chlorine, ethylene, fluorine, fluorocarbons, hydrogen chloride, hydrogen sulfide, krypton, LP-gases, medical gases, methane, nitrogen, nitrous oxide, oxygen, vinyl methyl ether, and xenon. An extremely important section of the book describes vital materials available nowhere else in book form - the most widely applicable safety standards for compressed gas handling and for container construction, developed by the compressed gas industry itself. These standards are the result of many years of research conducted by the Compressed Gas Association. Special attention has been given to recent advances, as in the book's coverage of cryogenic liquid gases used so spectacularly in the aerospace industries. The appendices contain valuable material summarizing the regulations pertaining to compressed gas shipment and storage that have been adopted by states throughout the United States.

HANDBOOK OF THERMAL DESIGN DATA FOR MULTILAYER INSULATION SYSTEMS - Coston, R. M. - Lockheed Missiles and Space Company - Prepared for NASA-Marshall Space Flight Center, Contr. No. NAS 8-20353, NASA CR-87485 (June 1967) 207 pp 71 fig 5 tab 115 ref.

The prime intent in assembling the data presented in this volume is to provide a sourcebook of consistent data on thermal and physical properties of multilayer insulations so that the data can be applied in mathematical thermal models of cryogenic propellant tankage.

The second intent of this report is to provide readily available design data for general usage by designers and thermal analysts of cryogenic space vehicles who will not necessarily employ the mathematical models. A variety of design data is presented, including thermal conductivity, specific heat, density, linear thermal expansion, weight per unit area, and maximum material operating temperatures.

Data presented here are divided into four major categories: (1) Gases; (2) Metals; (3) Fiberglass Laminates; (4) Multilayer Insulations; (4.1) Substrates; (4.2) Multilayer-Insulation-System Materials; (4.3) Multilayer Composites; (4.4) Attachment Methods; (4.5) Test Methods.

THERMAL INSULATION SYSTEMS - NASA Tech. Utiliz. Rept., NASA SP-5027 (1967) - 148 pp 77 fig 10 tab 101 ref.

The purpose of this survey is to summarize, and thus make more accessible to industry, data on the performance of thermal insulations and systems combining such insulations, particularly for cryogenic applications, and

to indicate opportunities for improvements. Included in this work are chapters on: Applications of Thermal Insulations; Principles of Thermal Insulation Systems; Cryogenic Insulation Systems; Structural and Non-insulating Cryogenic System Components; High-Temperature Thermal Protection Systems.

BOILING HEAT TRANSFER FOR OXYGEN, NITROGEN, HYDROGEN, AND HELIUM - Brentari, E.G., Giarratano, P.J. and Smith, R.V. - Natl. Bur. Standards Tech. Note 317 (Sept. 1965) 119 pp 83 fig 7 tab 110 ref.

This study has been conducted to provide an orderly examination of the information relative to boiling heat transfer for four cryogenic fluids. The general approach has been to examine experimental data with respect to the predictive correlations which would appear to have probable success and which would be likely to be used by design engineers. These correlations were graphically and statistically compared. The results are discussed, and when it appears a best or acceptable recommendation can be made, computation aids for designers are included. These aids are in the form of graphical presentations for preliminary studies and equations for computer studies. The authors have also indicated the apparent limits for the use of these correlations, when possible. The effect of many variables which would often be significant are not included in the predictive correlations. The influence of these variables is discussed in a separate section on boiling variables.

BOILING HEAT TRANSFER FOR CRYOGENICS - Seader, J.D., Miller, W.S. and Kalvinskas - Rocketdyne - NASA CR-243 (June 1965) NASA Contr. No. NAS 8-5337 117 pp 47 fig 9 tab 154 ref.

An extensive survey of available information from the literature on heat transfer to boiling hydrogen, nitrogen, and oxygen is presented. Included are a bibliography of available experimental data, a summary of boiling theory, graphical comparisons of the experimental data, and compilations of pertinent physical properties for each of the three cryogenic fluids. Based upon the results of the survey, recommendations are made for a test program to provide critical information where data are lacking.

REVIEW OF STATIC SEALS FOR CRYOGENIC SYSTEMS - Robbins, R.F. and Ludtke, P.R. - J. Spacecraft Rockets 1, No. 3, 253-59 (May-June 1964) 6 fig 1 tab 55 ref.

Although cryogenic techniques have advanced rapidly, little effort has been devoted to standardization of seals for cryogenic systems. This paper surveys and evaluates various demountable, low-temperature static seals. Rectangular-sectioned gaskets of Teflon or Kel-F, or of soft metal, or with plastic sealing surfaces can be used, but gaskets require heavy flanges. O-rings of rubber, Viton, and neoprene have been used successfully down to 20°K with high-initial-loading designs. However, metallic O-rings can be used, but they are hard and require good surface finishes, ≥ 16 rms. Solid O-rings of soft metals (e.g., indium, lead, copper, and aluminum) can seal to 10^{-5}

atm-cc/sec/linear-in. Pressure-actuated seals of C, U, V, or W configuration are said to be superior to all others for cryogenic space systems (a table of available types is given); they use lightweight flanges and will follow flange deflections; they are expensive, and small leak paths can develop, but they have proved reliable in the field. Temperature-actuated seals (favorable thermal contraction relationships) also show promise but have not been used extensively.

BEARINGS AND SEALS FOR CRYOGENIC FLUIDS - Scibbe, H. W. - NASA - Lewis Research Center, Cleveland, Ohio - NASA TM X-52415 (March 1968) - [Technical paper presented at SAE Fuels and Lubricants Meeting, Cleveland, Ohio, Nov. 13 1967].

Bearings and seals in rocket engine turbopumps operate directly in the cryogenic propellant. Special design and lubricating techniques are required since ordinary oils and greases become glasslike solids at these extremely cold temperatures. The bearing load carrying surfaces are lubricated by thin transfer films. The lubricant is provided by the bearing cage which is usually fabricated from a self-lubricating Teflon compound. Material compatibility and wear are the important factors for face contact seals in these cryogenic fluids. Carbon, normally used in seal nosepieces, has violent reactions when run in liquid fluorine, an extremely chemically active fluid. Wear at the rubbing contact is minimized when the seal is designed for positive face separation with acceptable leakage.

INTRODUCTION TO CRYOGENICS - Vance, R. W. - Machine Design 36, 169-192 (Oct. 8, 1964).

The principal engineering problems associated with cryogenics, including the fundamental relations, theories, equations and concepts, have been described in detail by many authorities. This article highlights some of these considerations and points the way toward practical solutions to selected design problems.

INTRODUCTION TO CRYOGENIC ENGINEERING - Reiff, D. D. - ASME Paper No. 64-WA/PID-8 (Nov. 1964) 12 pp 7 fig 11 ref.

The purpose of this report is to introduce a few of the essential disciplines in cryogenic engineering. The basic principles of liquefaction of gases and low-temperature heat transfer and insulation are briefly treated. The discussion of storage, transfer, and handling of cryogenic fluids has been slanted to hydrogen; however, the basic principles of cryogenic technology are embraced. In addition, mobile ground-service equipment is discussed briefly.

THE STORAGE AND HANDLING OF CRYOGENIC LIQUIDS - Zenner, G. H. - Progress in Cryogenics 2, 3-39, Academic Press Inc., New York (1960) 24 fig 6 tab 51 ref.

Included in the contents of this article are sections on the properties of liquids O₂, N₂, A, CH₄, F₂, H₂, He, storage and transport containers (stationary, truck, tank

car), converters, meters, pumps (rotary and reciprocating), transfer methods and piping design.

SAFE HANDLING OF CRYOGENIC FLUIDS - Neary, R. M. - Chemical Section, National Safety Congress, Chicago, Ill., (Oct. 16, 1961) paper 20 pp 8 ref.

Cryogenic fluids have been handled safely by a few companies since the early part of the century. Their use, and the number of people handling them, have increased tremendously in recent years. To ensure safety, old and new users alike require a thorough understanding of the properties of these fluids and a willingness to make use of reasonable safeguards in handling them. Since handling small quantities presents some safety considerations different from handling larger volumes, these subjects will be covered in separate sections of this paper.

AIR-CONDENSING CRYOGENIC FLUIDS - Neary, R. M. - Union Carbide Corp., Linde Division - Paper presented at National Safety Congress (Oct. 31, 1963).

With the recent advances in the missile and electronics industries and of numerous research applications, such as Telstar and space chambers, liquefied hydrogen and helium are fast becoming important cryogenic fluids. Unlike the more common cryogenic fluids, namely oxygen and nitrogen, the properties of liquefied hydrogen and helium are unusual and, as a result, special equipment and handling techniques are required. This paper briefly summarizes the properties, design of equipment, and practical procedures for the safe and efficient handling of these fluids.

AIR-SOLIDIFYING CRYOGENIC FLUIDS - Neary, R. M. - ASME Paper No. 64-WA/SAF-1 (Sept. 1964) 8 pp 8 fig 1 tab 8 ref.

With the recent advances in the missile and electronics industries and of numerous research applications, liquefied hydrogen and helium are fast becoming important cryogenic fluids. Unlike the more common cryogenic fluids, namely liquid oxygen and nitrogen, liquefied hydrogen and helium are capable of solidifying air. This and other properties make it necessary to handle these products with different equipment and procedures. Personal protective equipment is reviewed. The design of small, medium and large tankage is reviewed briefly. Special emphasis is placed on design and safety devices for small liquefied helium containers commonly used in laboratories because several serious accidents have resulted from solid air plugging of the vents.

CRYOGENIC SAFETY - Spencer, E. W. - J. Chem. Education 41 (Sept. 1964) 4 pp 5 fig 1 tab 14 ref.

A general discussion on the hazards, storage, and general precautions pertinent to the handling of cryogenic fluids. This article is a portion of the paper which appeared previously in the Journal of American Society of Safety Engineers.

FIRE-HAZARD PROPERTIES OF FLAMMABLE LIQUIDS, GASES AND VOLATILE SOLIDS - National Fire Protection Association, Rept. No. 325 (May 1960).

This tabulation of available data on the properties of flammable liquids and other materials listed is sponsored by the NFPA Committee on Flammable Liquids. The table summarizes available data on the fire-hazard properties of more than 1000 substances, listed alphabetically by their chemical name. The values selected are representative figures deemed suitable for general use.

REVIEW OF FIRE AND EXPLOSION HAZARDS OF FLIGHT VEHICLE COMBUSTIBLES - Van Dolah, W., Zabetakis, M. G., Burgess, D. S. and Scott, G. S. - ASD TR 61-278 (April 1961) (See also subsequent annual supplements).

The prevention of fires and explosions involving the combustibles and oxidants likely to be found in flight vehicles requires a knowledge of the flammability and related characteristics of these materials. This is a compilation of the available characteristics data for a series of combustibles and oxidants of current interest (including fluorine, oxygen and hydrogen).

EXPLOSION AND FIRE HAZARDS ASSOCIATED WITH THE USE OF LOW-TEMPERATURE INDUSTRIAL FLUIDS - Burgoyne, J. H. - Trans. Inst. Chem. Engrs. (London) 185, CE 7-10 (Jan. - Feb. 1965).

Hazards of low-temperature fluids are considered under the headings of pressure bursts, gas explosion, fire, and explosion of liquid mixtures (e.g., of fuel and oxygen). The literature on these subjects is reviewed and an attempt is made to distinguish areas where gaps in knowledge exist and further systematic research should be rewarding. It is concluded that gas explosion properties at low temperatures and the explosibility of low-temperature liquid mixtures merit considerable attention.

SPARKING CHARACTERISTICS AND SAFETY HAZARDS OF METALLIC MATERIALS - Bernstein, H. and Young, G. C. - NAVORD Rept. 5205, Tech. Rept. NGF-T-57 (April 1957).

This report is a survey of the sparking characteristics and safety hazards of metallic materials. The fundamentals of sparking theory and methods of spark testing are presented. The ignition hazards associated with sparks are discussed. Attention is called to an alternate and possibly more significant source of ignition - impacts. The data indicate that sparks and impacts can result from the use of "non-sparking" materials. The authors conclude that no benefit is gained by employing non-sparking hand tools in place of steel to prevent explosions.

RECENT ADVANCES IN CRYOGENIC ENGINEERING - Jacobs, R. B. - ARS Jour., 245-51 (Apr. 1959) 56 ref.

The point to be emphasized is that cryogenic engineering is a synthesis of the older branches of engineering (mechanical, chemical, electrical), and therefore does not involve new basic principles. The discussion presented here has avoided those areas where usual engineering ex-

perience is directly applicable. An attempt has been made to discuss situations where the unwary may go astray and where the cryogenic engineer has evolved new techniques. In addition, an attempt has been made to indicate the location of data and information applicable to new materials and situations, as well as that applicable to the familiar materials and situations in the low temperature regions.

PROBLEMS IN CRYOGENICS - Nesbitt, L. B. - General Electric Co., Schenectady, N. Y. - ASME Paper No. 63-WA-288 (Nov. 1963) 4 pp 1 fig.

A major impediment in the growth of cryogenic engineering as a productive segment of the engineering sciences has been the many real and imaginary problems whose solutions are now available. The author points out the pitfalls in the study of cryogenics and indicates a sound approach to cryogenic experimentation and development.

THERMAL PROBLEMS PECULIAR TO CRYOGENICS IN SPACE - Adelberg, M. and Schwartz, S. H. - SAE Paper No. 670588 (June 1967) 10 pp 9 fig 3 tab 24 ref. - [Published in Proc. of the SAE Aerospace Systems Conference, Los Angeles, Calif.]

Thermal problems peculiar to cryogenics stored in a reduced gravity environment are discussed along with some techniques considered for their solution. Included are gravitational effects upon transient and steady state nucleate boiling, heat transfer and temperature fluctuation and meniscus displacement and distortion. To place these problems in proper perspective, a brief survey of the various heat transfer modes is presented. One device, which appears to warrant serious consideration, is the thermodynamic separator and its operation is briefly described.

To illustrate the importance of certain problems peculiar to reduced gravity, a number of numerical examples are presented.

HANDLING LIQUID PROPELLANTS - Dachs, L. L. - Space/Aeronautics 46, 77-84 (Oct. 1966).

Beyond obvious fluid management problems, liquid propellants spawn sneaky, second order effects like cavitation, oscillation and zero-g "migration". Equipment configuration is the key to knocking down bubble population; baffles and the like arrest sloshing; expulsion devices help the orbital restart.

CRYOGENIC INSTRUMENTATION - Angerhofer, A. W. - Air Reduction Co., Inc. - Control Engineering 12, 67-73, 77-84 (1965) (A two-part article).

Because of their low temperatures and high volatility, some unique measurement problems arise in the use of cryogenic fluids. Conventional sensors can solve most of these problems, but satisfactory operation requires care in application and a knowledge of the complications that can occur. The first article discusses special cryogenic prob-

lems in temperature and level sensing; the second article covers fluid flow and analysis instruments.

CRYOGENIC THERMOCOUPLE THERMOMETRY - Sparks, L. L. and Powell, R. L. - Measurements and Data 1, 82-90 (March - April 1967)

Commercially available low-temperature thermocouple wire from all major U.S. manufacturers has been exhaustively tested to determine the inhomogeneity and interchangeability characteristics of the wire. Spot calibrations between liquid-helium and liquid-nitrogen temperatures, and between liquid-nitrogen and ice temperatures, show that the NBS interim low-temperature tables are sufficient for most engineering and scientific requirements. Preliminary work on several gold-iron alloys indicates that these alloys will allow more accurate thermoelectric temperature measurement in the liquid-helium/liquid-hydrogen temperature range. Research, now well underway, will lead to establishment of standard thermocouple tables for temperatures from liquid helium up to 0°C for all of the cryogenically useful commercial thermocouple alloys.

MASS FLOWMETERS IN CRYOGENIC SERVICE - Alspach, W. J., Miller, C. E. and Flynn, T. M. - Paper presented at 1966 ASME Flow Measurement Conf., Pittsburgh, Pa. - (Published in Flow Measurement Symposium) 23 pp 27 fig 29 ref.

This paper concerns cryogenic fluid mass flow measurement by a variety of techniques, including those that are available and those that are being developed. Attention is given to the principle of operation, performance results, and operational and design characteristics.

The problem of cryogenic fluid flowmeter calibration is examined and discussed with reference to available facilities, techniques, limitations, and accuracy. The problem of calibration for special cryogenic applications, such as slush hydrogen and cold gases, where no calibration facilities are available is also examined. Inferred calibration from a substitute fluid calibration or design practices is also reviewed.

To improve the mass flow measurement of cryogenic fluids, discussions are directed towards measurement technique selection, density measurements for inferential mass systems, improvements in volumetric flow measurements, improvements in direct measuring mass flowmeters, and improvements in calibration.

CRYOGENIC FLOW MEASUREMENT - FLOW METERS - Bucknell, R. - Cryogenic Eng. News 1, 28-30, 43 (April 1966).

The author presents a discussion of flow meters and their characteristics in cryogenic service.

FLOW MEASUREMENT OF CRYOGENIC FLUIDS - Close, D. L. - Instruments and Control Systems 41, 109-114 (Feb. 1968) 7 fig 4 tab 15 ref.

The fundamentals of flow measurement of ambient temperature fluids can be applied to cryogenic liquids, which are typically colder than -150°C. There are three special

concerns in cryogenic flow measurement regardless of meter type: (1) the condition of the liquid to be metered; (2) the materials and construction of the primary element; (3) the safe installation of that element.

LARGE SIZE CRYOGENIC TURBINE TYPE FLOWMETER TECHNOLOGY - Deppe, G. R. - Aerojet-General Corp. Rept. No. 8800-60, - NASA CR 54810 (June 1966) 45 pp 14 fig 3 tab 11 ref.

The procurement, calibration, facility installation, and use of turbine flowmeters for the very large liquid oxygen/liquid hydrogen components of the M-1 Rocket Engine are described. Also, construction details of one turbine flowmeter and limited calibration data for several turbine flowmeters are given. The relative capabilities of the nation's major testing organizations are presented along with facility recommendations.

COOLDOWN OF LARGE-DIAMETER LIQUID HYDROGEN AND LIQUID OXYGEN LINES - Commander, J. C. and Schwartz, M. H. - Aerojet-General Corp. - Rept. No. 8800-54, NASA CR 54809 (April 1966) 56 pp 22 fig 6 tab 8 ref.

This report concerns the analytical study and operational experience associated with the large cryogenic propellant systems used in Test Zone E of the M-1 test complex. The techniques applicable to initial chilldown of large diameter cryogenic piping systems are discussed; major consideration is given to liquid hydrogen and liquid oxygen propellant systems that utilize the cryogenic boil-off gases at a predetermined rate to effect system chilldown. The use of this technique reduces or eliminates the problem of thermal stress concentrations in the system materials during chilldown.

The principal areas of discussion include:

- A. Various methods considered for chilldown of cryogenic piping systems.
- B. Analysis of thermal stress at selected points in liquid hydrogen and liquid oxygen piping systems where cross-section transitions produce maximum thermal gradients.
- C. Analysis of system chilldown characteristics when cold gaseous cryogens are utilized to effect the initial reduction in system temperatures, including predictions of time in relationship to equilibrium temperature for systems exposed to various combinations of gas flow rate and initial temperature.
- D. Analysis of the effects caused by cold shocking on critical cross-section transitions of the 18 inch liquid hydrogen discharge line which have stabilized at equilibrium temperatures of 260 R, 160 R, and 60 R.

COOLDOWN OF CRYOGENIC TRANSFER SYSTEMS - Liebenberg, D. H., Novak, J. K. and Edeskuty, F. J. - AIAA Paper No. 67-475 (July 1967) 9 pp 8 fig 3 tab 16 ref.

Operation of a cryogenic system is frequently complicated by the transient process of cooling it to its equilibrium operating temperature. A knowledge of the time and amount of cryogen necessary to cool the line is valuable since sufficient cryogen storage must be provided to

complete the cooling of a large line. It is also necessary to cool the line in a manner which will not result in damage to the line or to associated components. Improper cooldown procedures can produce bowing (a distortion of the pipe caused by uneven cooling around the pipes circumference), unacceptable pressure and flow surges if the flow is improperly initiated or limited, unacceptable stresses in components if temperature gradients are too large, or can, through uneven cooling, cause binding in moving parts, such as pumps, mechanical interference of components, etc. Specific rules for eliminating these problems cannot be given, but general suggestions for avoiding the problems are presented.

FLEXIBILITY CONSIDERATIONS FOR THE DESIGN OF CRYOGENIC TRANSFER LINES - Flieder, W. G., Smith, W. J. and Wetmore, K. R. - Advances in Cryogenic Engineering 5, 111-19 (Proc. of 1959 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) Paper B-5, 9 fig. 5 ref.

In conventional piping systems, sufficient uniform thermal cooldown flexibility is obtainable by the use of expansion loops. Uniform thermal cooldown occurs when the pipe is full of liquid and the entire pipeline has cooled to liquid temperature. In the case of missile systems employing cryogenic transfer lines, space and cooldown weight limitations may preclude the use of expansion loops for achieving flexibility. In addition, the uninsulated thin-walled transfer piping is subject to temperature gradients of a scale not usually encountered in process piping systems, and as a consequence of these temperature gradients the phenomenon of pipeline bowing occurs. Bowing is the tendency of the centerline of the pipe to be deformed into an arc.

A brief analytical study is presented to show how flexibility for cryogenic lines may be obtained by the use of expansion loops and expansion joints that satisfy both the requirements for uniform thermal cooldown and bowing considerations. Finally, the liquid oxygen transfer piping developed for the Atlas missile launching complex is presented and special features of this system are explained.

CRYOGENIC PIPING SYSTEM DESIGN CONSIDERATIONS - Jacobs, R. B. - Heating, Piping Air Conditioning 32, 130-40, 142-56 (1960) (A two-part article).

Piping systems for transporting liquefied gases are not basically different from those for the more familiar fluids. But, some differences must be considered by the designer because of the temperature range encountered - room temperature down to almost absolute zero or -459.6°F . The topics presented (in the first article) include properties of materials and insulation techniques. How low temperatures affect the fluid-mechanical design, selection of pumping equipment, heat exchanger design, piping system instrumentation, and the fabrication and installation of the piping system are presented in the second article.

FUNDAMENTALS OF LOW TEMPERATURE PIPING DESIGN - Surdi, V. L. and Romaine, D. - The M. W. Kellogg Co. - Hydro. Proc. and Petr. Ref. 43, 116-24 (June 1964).

While the design of piping for low temperature is, for the most part, the same as for above ambient temperature, there are two basic conditions that must be given special consideration.

First, the cost of refrigeration increases as the temperature is reduced. From this it is clear that low temperature insulation and insulation techniques merit special design consideration, and, as is shown in this paper, such consideration leads to a number of concepts that differ from conventional warm piping.

Second, carbon steel and gasket materials become brittle at lower temperatures. For operation below -50°F , it is necessary to choose either nickel steels, stainless steel, copper, or aluminum as the material of construction. While each of these materials serves very well in certain situations, each nevertheless has certain limitations that require special design consideration. Gasket materials become brittle and are generally less effective at low temperatures. Designing flanges and joints to avoid leaks takes on a new significance in low temperature plants where vessels, piping, and valves frequently are made of dissimilar metals with attendant differences in coefficients of thermal expansion.

CRYOGENIC STORAGE VESSELS - Chicago Bridge and Iron Co. - Publication G-50-64 - 15 pp 18 fig 4 tab.

The information contained in this bulletin deals primarily with storage vessels. Included are sections on insulation systems, vessel geometry and design requirements, materials of construction, fittings, and foundations.

HOW TO SPECIFY LOW TEMPERATURE STORAGE VESSELS - Zick, L. P. and Clapp, M. B. - Chicago Bridge and Iron Co. - Hydro. Proc. and Petr. Ref. 43, 125-32 (June 1964).

Greater exposure to brittle fracture at full stress is the most significant difference between vessels used for storage at refrigerated temperatures and storage at atmospheric temperature. Therefore, the selection of notch tough material is extremely important for refrigerated storage vessels. Notches should be eliminated in the design details and construction of parts carrying high stress, insofar as possible. Unavoidable stress concentrations are better tolerated by notch tough materials.

This article is based upon the current state-of-the-art, but the reader should remember that a precise evaluation of the ductile versus brittle behavior of materials used in storage vessels operated at refrigerated temperatures is not clearly defined. Nevertheless, certain levels of toughness which may be determined by tests will result in safe structures.

DESIGN OF CRYOGENIC STORAGE TANKS FOR INDUSTRIAL APPLICATIONS - Marsh, H. W. - Am. Soc. Testing Materials, Spec. Tech. Publ. No. 302 (March 1962) pp 172-83, 4 fig 1 tab 4 ref.

This is a discussion of the facets of cryogenic storage tank design directed toward those who have only limited experience in the field. Design considerations as to cost, suitability of materials for the temperatures and pressures involved, configuration of inner vessels and jackets, support systems, and types of insulation, evacuated and nonevacuated, for both shop-built and field-erected vessels are discussed in brief. The potential requirements for cryogenic storage for industrial applications are listed. Military applications for both ground and air-borne use are excluded as these involve unusual design conditions foreign to the usual industrial installation.

LARGE CRYOGENIC STORAGE VESSELS - Adams, L. - Pittsburgh-Des Moines Steel Co. - ASHRAE Jour., 48-9 (July 1964).

This article summarizes data on the size, shape, and design of large capacity, field-erected vessels for the storage of cryogenic fluids, and includes information on metals, thermal insulation, heat leak, and some pertinent physical properties of the products stored.

CONSTRUCTION OF HIGH PRESSURE CRYOGENIC VESSELS - Sangdahl, G. S. and Wilson, L. C. - Chicago Bridge and Iron Co. - ASME Paper No. 64-PET-17 (Sept. 1964) 12 pp 8 fig 4 tab 11 ref.

The large increase in the use of oxygen, nitrogen, hydrogen, methane and other gases for industrial purposes has greatly increased the need for storage of these gases. This can most efficiently and safely be done as a liquefied gas at a temperature below the boiling point of the gas and at atmospheric pressure. Special applications may require high pressure storage. This article describes the many factors which must be considered in the construction of high pressure cryogenic vessels - such as: design, material selection, forming, welding, heat treatment, inspection, and cleaning.

THE PAST, PRESENT, AND FUTURE OF METALS FOR LIQUID ROCKETS - Lucas, W. R. - Metals Engineering Quarterly 6, 58-62 (Feb. 1966).

The utilization of metals in liquid-propelled rocket systems is discussed in terms of the unique environments in which performance is required. Weldability, toughness at cryogenic temperature, stress-corrosion resistance, and chemical compatibility with propellants are identified as important criteria in the selection of metals for these applications. Aluminum has been the predominant metal in liquid rocket structures, and its use has paralleled aluminum alloy development in this country. Recent achievements in the development of new wrought aluminum alloys, a new aluminum casting alloy, and other lightweight materials are discussed. Composite materials are projected as the materials of the future because they afford the possibility of increasing the strength-to-density ratio

by the reduction of density rather than by the increase of strength and the possibility of utilizing simultaneously the most favorable properties of two or more materials.

LOW TEMPERATURE AND CRYOGENIC STEELS; HOW TO CHOOSE, USE, AND WELD THEM - Anonymous - Special Report - Welding Design and Fabrication, 51-73 (April 1964).

In this report information is provided on which steels are proving to be best for each low temperature level; what welding electrodes and methods are recommended; which ASME pressure vessel codes apply; and what ASTM designations are used for each steel grade.

ROCKET PROPELLANTS - Armed Services Tech. Inform. Agency, Arlington, Va., Bibliography Rept. AD 233 500 (March 1960) 56 pp.

The bibliography is a compilation of approximately 292 abstracts of unclassified reports on liquid and solid propellants which were added to the ASTIA collection from 1953 to 15 Feb. 1960. The first part of the bibliography includes separate sections dealing with general information on solid rocket propellants and on liquid rocket propellants. The second part of the bibliography includes sections for each of the following compositions used in rocket propellants: (1) acetylenes, (2) amines, (3) ammonia, (4) ammonium perchlorate, (5) boron hydrides, (6) ethylene oxide, (7) fluorine, (8) hydrazines, (9) hydrocarbons, (10) hydrogen, (11) hydrogen peroxide, (12) lithium compounds, (13) methanol, (14) nitric acid, (15) nitrogen, (16) nitrogen-fluorine compounds, (17) nitrogen oxides, (18) nitro-paraffins, (19) oxygen, (20) ozone, (21) perchloryl fluoride, and (22) thiophosphites. Abstract entries in each subdivision of the first and second parts are arranged alphabetically by corporate author, numerically by contract number and by date. Only final or summary reports were included in the bibliography, except when the final report was not received or when a progress report contained significant information which was not included in the final report. A bibliography of confidential entries on solid and liquid propellants (AD 315 500) was compiled separately.

ROCKET PROPELLANTS - Armed Services Tech. Inform. Agency, Arlington, Va., Bibliography Rept. AD 263 000 (Aug. 1961) 75 pp.

A list of references was prepared as a sequel to previous ASTIA bibliographies on rocket propellants identified as AD 233 500 (unclassified) and AD 315 500 (confidential). Citations are limited to unclassified documents cataloged by ASTIA from February 1960 to August 1961. A classified edition of the bibliography was also published as AD 325 000. Entries are arranged in two broad categories of references: liquid, hybrid, solid or non-conventional propellant systems, and specific chemical components of propellants.

CRYOGENICS AND LOW TEMPERATURE RESEARCH. AN ASTIA REPORT BIBLIOGRAPHY - Armed Services Technical Information Agency, Arlington, Va., ASTIA AD 271 000 (Feb 62) 865 refs.

This bibliography was presented by ASTIA in response to numerous inquiries concerning cryogenics and low temperature research. Citations are included for documents cataloged by ASTIA from 1953 through 1 February 1962, and are restricted to unclassified, unlimited references. The classified section of the bibliography appears separately as a secret document, identified as AD-327 650. References are arranged alphabetically by subject areas pertaining to low temperature research, instrumentation, and materials. These subject areas are further subdivided into more specific topics which include references on superconductivity, thermochemistry, temperature measurement and control, adhesives, elastomers, liquefied gases, lubricants, metals, and propellant research.

CRYOGENICS - AN INTERNATIONAL JOURNAL OF LOW TEMPERATURE ENGINEERING AND RESEARCH - Mendelssohn, K., Scott, R. B., and Weil, L. (Editors) Heywood and Company, Ltd., London (Distributed in U.S. and Canada by Plenum Press, Inc., New York)

The purpose of CRYOGENICS is to publish original papers on all aspects of low temperature research, engineering, and development. Each issue also features an invited survey article, written by an authority on the subject, as well as shorter technical notes, letters and book reviews. The complete texts of all papers appear in English, with abstracts printed in English, French, German, and Russian.

The many different trends of low temperature research and development today are not only intensified, they are also divergent. However, all share the same cryogenic

methods and techniques and all must rely on the same basic research and engineering data. Concentration of relevant information on these subjects in one journal should be invaluable to all those working in the field. It is the aim of CRYOGENICS to save time and labor of researchers and engineers in the field by providing them with significant and current information in one convenient publication.

CRYOGENIC ENGINEERING NEWS - (A controlled circulation publication printed monthly by Business Communications, Inc., Cleveland, Ohio).

A publication concerning the production and use of ultra-low temperatures.

CRYOGENIC INFORMATION REPORT - (Published by Technical Economics Associates, Estes Park, Colo.).

A monthly survey, bringing together information on the developments and activities in the various areas of the cryogenic field.

CRYOGENIC TECHNOLOGY - JOURNAL OF THE CRYOGENIC SOCIETY OF AMERICA - (Published bi-monthly by Cryogenic Technology Publications, Inc., Bel Air, Los Angeles, Calif.).

CRYOGENIC TECHNOLOGY is an official publication of the Cryogenic Society of America, devoted to the various technical and practical aspects of the several branches of cryogenics.

CRYOGENIC DATA CENTER

Since the purpose of this report is to provide summary information concerning fluid and solid material properties and practices relating to selected cryogenic propellants, it may be considered useful to indicate something about the primary world source for such information - the National Bureau of Standards Cryogenic Data Center. The Center is a Section in the Cryogenics Division of the National Bureau of Standards (Boulder, Colorado Laboratories) and is divided into two principal groups, the Data Compilation Unit and the Documentation Unit.

The Data Compilation Unit is engaged in the critical evaluation and compilation of thermodynamic and transport properties of cryogenic fluids and fluid mixtures and cryogenic properties of solids. Tables and charts of property data, based on selected values from the scientific literature, are compiled for wide ranges of temperature and pressure. As a participant in the National Standard Reference Data System program, the Data Compilation Unit is recognized as a national authority for data on the properties of materials at cryogenic temperatures, and thus many of the tasks undertaken by this unit are organized to produce data tables suitable for entry into the NSRDS.

The Documentation Unit's functions complement the activities of the Data Compilation Unit. These functions are: 1) to maintain an awareness of current publications and reprints of cryogenic interest, 2) to acquire and catalog such literature as needed by the Data Compilation Unit, other laboratory staff, and for completing the Data Center's files, 3) to code the pertinent literature in depth for storage and retrieval, and 4) to develop an operative mechanized bibliographic and indexing service for the comprehensive retrieval of information and data in specific subject areas as needed. This unit also handles the distribution of all the Cryogenic Division's publications and announces availability of new materials periodically.

Of possible interest to the readers of this document are the various services available to the public from the Cryogenic Data Center:

Literature Searches. Nearly 60,000 accessions of cryogenic literature have been entered into the Data Center's system. Approximately two-thirds of these (on the properties of materials, cryogenic equipment and processes) have been processed for machine searching. Custom bibliographies are prepared for specific subjects or for broad subject areas; indexing follows from the nature of search queries and can be quite detailed. Simple searches can be made for as little as \$25.00 with more extensive searches at a proportionately higher cost. The feasibility and estimated cost of a search can be obtained from the Data Center.

Current Awareness Service. An awareness of publications and reports of cryogenic interest is maintained by the regular review of nearly two hundred periodicals cover to cover, by a weekly review of the "Current Contents" service, by reviewing some fifteen abstract journals, and by noting references in cryogenic documents. Weekly lists of new literature of cryogenic interest are prepared and distributed to subscribers at a subscription price of \$15.00 per year for 52 issues. A subscription may be obtained simply by ordering from the Cryogenic Data Center.

Announcements of Cryogenics Division Publications and Reports. Announcements and abstract cards of new literature evolving from the Cryogenic Division's research programs are sent to more than 4000 persons and institutions periodically. Nearly five hundred separate items of literature are now available. Request can be made for inclusion on the mailing list by writing to the Cryogenic Data Center. Distribution of the available literature is now being handled by the government Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia 22151. Ordering information is included with the announcements.

Anyone desiring information concerning the various services described above may write to the

Cryogenic Data Center
National Bureau of Standards
Boulder, Colorado 80302

or telephone

(303) 447-1000, ext. 3834

II. RECOMMENDED MATERIALS AND PRACTICES FOR USE WITH LIQUID OXYGEN

- A Compilation from the Literature with Annotated Bibliography -

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BIBLIOGRAPHY

1. HAZARDS

1.1 Health - The health hazards of liquid oxygen arise from its very low temperature. If liquid, or cold gaseous, oxygen contacts the skin, damage resembling burns can result. The extent of such damage can range from relatively minor burns to complete embrittlement and destruction of exposed tissue. The immediate effects of freezing by liquid oxygen can be minimized by soaking affected parts in tepid water. Extensive burns require prompt medical attention.

Oxygen gas will not cause toxic effects in propellant operations, except that inhalation of the very cold gas may cause some upper respiratory irritation.

1.2 Fire - Liquid oxygen will not burn but vigorously supports combustion. Its low temperature causes cooling and freezing of liquid fuels if both liquids are brought together. Such mixtures are shock sensitive and are capable of reaction with the violence of a detonation.

Liquid oxygen is capable of saturating normal clothing and rendering it flammable. Workers must not smoke or strike fires in oxygen storage or handling areas or while wearing clothing saturated with oxygen, since the clothing may retain a high concentration of oxygen for a considerable period of time.

Two types of combustion reactions may occur depending on conditions of mixing and ignition. Intensity of combustion reaction is dependent on the type of fuel involved. Although the mixing of fuel and liquid oxygen may be accomplished without fire, a detonation occurs upon ignition or

mechanical shock excitation. Where combustion is initiated prior to or concurrently with fuel-oxygen contact, flare type combustion occurs, accompanied by repeated explosions.

Putting out fires involving liquid oxygen requires procedures varying with the type of fire conditions. Where the fire results from a leak or flow of liquid oxygen onto wood, paper, waste, or similar combustible material, first stop the flow if possible. For small spills, or after the leak or flow of liquid oxygen has been stopped, use enough water to put the fire out quickly. Where the fire involves liquid oxygen and liquid fuels, control it as follows:

(a) When liquid oxygen leaks or flows into large quantities of fuel, shut off the flow of liquid oxygen, and put the remaining fuel-fire out with Class B fire fighting agents.

(b) When fuel leaks or flows into large quantities of liquid oxygen, shut off the fuel flow.

(c) When fuel and liquid oxygen have been mixed, or are mixing, but not yet burning, isolate the area from ignition sources and get out quickly, allowing the oxygen to evaporate. Where large pools of water-soluble fuel are present, use water to dilute the fuel and to reduce the intensity of the fire. This method cannot be used with fuels which do not mix with water. Do not use on fires in deep pools of liquid oxygen as this causes more rapid release of gaseous oxygen. Appropriate extinguishing agents may be used to put out fuel fires after the oxygen has evaporated.

NOTE: Mixtures of liquid oxygen and fuels present an extreme detonation hazard. Such materials as wood pulp, cotton, lampblack, hydrocarbons, metal powders, sulfur

and coal dust are often used in combination with liquid oxygen as a relatively cheap explosive; the latter combination produces an effect similar to 40% nitroglycerine dynamite. It seems likely that almost any combustible material might detonate when in contact with liquid oxygen. Several incidents may illustrate this: A leak developed in a pipe joint in a liquid oxygen line and the liquid flowed onto an asphalt-paved surface. When a workman attempting to repair the leak struck the joint, the impact was transmitted from the joint to the pavement below and the pavement detonated. A similar incident has been reported where gravelled asphalt detonated when a man walked across an area where liquid oxygen had previously been spilled.

1.3 Explosion - All materials that will burn, especially rocket fuels, present an explosion hazard when mixed with liquid oxygen. Such mixtures can usually be exploded by static electricity, mechanical shock, electrical spark, and similar energy sources. Under most conditions, the ordinary burning of rocket fuels or other combustible materials, when mixed with liquid oxygen, may progress to a detonation.

Liquid oxygen forms high concentrations of oxygen gas from spills or leaks. During transfer operations large volumes of gas are formed due to "boil-off." In confined areas, gaseous oxygen can form mixtures with fuel vapors that can be exploded by static electricity, electrical spark, or flame.

Pressure rupture can occur when the liquid is held in a closed system with no refrigeration. Oxygen cannot be maintained as a liquid if its temperature rises above the critical temperature (-181°F) regardless of confining pressure. Liquid oxygen trapped between valves can cause violent rupture of the pipe or tube, while loss of refrigeration can cause a storage tank to rupture if the pressure is not relieved by suitable devices. Loss of vacuum in vacuum-jacketed tanks can cause increased evaporation and overload the normal venting system, resulting in high pressures.

All sources of ignition or heat must be kept away from oxygen transferring and servicing operations and from areas where spills have occurred. All tanks and equipment must be provided with proper grounding to remove static electricity. It is absolutely essential to keep combustible or other reactive materials to a minimum in liquid oxygen storage and handling areas. Porous combustible materials such as clothing may retain hazardous quantities of gaseous oxygen, creating a dangerous fire hazard.

Pressure rupture of equipment can be avoided by checking all parts of the oxygen system to see that refrigeration and/or vacuum jacketing is maintained. Closed systems and "dead ends" must be avoided unless properly protected with pressure relief valves and blow-out discs. Such devices protect the system in the event of refrigeration or vacuum failure. Blow-out discs, or their equivalent, are also required on vacuum-insulated equipment jackets.

2. SAFETY MEASURES

2.1 General - All hazardous operations or experiments involving the handling of liquid oxygen shall be performed

by two or more persons working in a group. Trained supervision of all potentially hazardous activities involving liquid oxygen is essential.

2.2 Personnel Education - The following subjects shall be explained to all persons concerned with liquid oxygen handling, transfer, and storage:

- (a) Nature and properties of oxygen in both the liquid and the gaseous phases.
- (b) Approved materials which are compatible with liquid oxygen.
- (c) Proper equipment and its operation.
- (d) Use and care of protective equipment and clothing.
- (e) Safety, self-aid and first-aid instructions.

2.3 Personal Protection - The principal hazards associated with the handling of liquid oxygen are fire and the extremely low temperature of the liquid.

For hand protection, gauntlet type gloves which can be easily and rapidly removed are satisfactory. These gloves may be either of asbestos or degreased chrome leather with an inner liner of impermeable material. For protection of the feet, leather shoes which can be readily removed should be used. These can be high top or low top; the choice depends entirely on the area in which the individual will be working. If high top boots are used, pants legs will be outside the boot tops. Where soles of shoes have been exposed to the liquid, the footwear should be removed immediately to prevent delayed frostbite.

Head and face protection requires the use of acid-type goggles (preferably) or a face shield to stop splashes. Flame resistant and static-free clothing should be worn by personnel conducting hazardous operations or experiments with liquid oxygen because of the fire hazard. In addition, an apron of approved material shall be worn if the liquid is being handled in an open system.

3. TRANSFER AND STORAGE

3.1 General - Liquid oxygen must be stored in containers (either fixed or mobile) of approved design, materials, and construction.

Storage, transfer, and test areas must be kept neat, and free from combustibles. These areas must be inspected frequently.

An adequate water supply or fire extinguishers must be available for combating fires. Approved deluge-type personnel showers should be properly located for immediate use in an emergency.

3.2 Materials - The ability of materials to maintain satisfactory physical properties and to withstand thermal stresses caused by large temperature changes is of prime importance.

3.2.1 Metals - The ferrous alloys, except the austenitic chromium-nickel alloys, lose their ductility when subjected to the low temperatures of liquid oxygen and depending on their form and the application to which they are applied they may become too brittle for use with liquid oxygen. Metals suitable for this service are aluminum, copper,

nickel, and most of their alloys, as well as the "300 series" austenitic stainless steels.

3.2.2 Non-Metals - Non-metals which are suitable for use with liquid oxygen are given in several of the references provided in the Bibliography. A rather extensive list of materials to be used for gaskets, packaging, sealants, lubricants, solvents, etc., is now available; however, use is occasionally qualified by the intended application - a fact which points to the desirability of reviewing the original information source concerning material compatibility with liquid oxygen. Applications involving mechanical impact are of particular concern.

3.3 Equipment - Liquid oxygen handling equipment shall be degreased by washing with approved grease-removing solvents before being used. Equipment taken out of service for maintenance or modification shall be inspected and cleaned before being returned to service.

Liquid oxygen may be stored in either fixed or mobile tanks of approved design and materials. Storage and shipping containers designed for non-cryogenic fluids shall not be used in this service. Storage tanks shall be proof-tested prior to service in accordance with the provisions of applicable ASME, ASTM, or ICC specifications for pressure vessels. Containers for shipment, storage, and transfer of liquid oxygen should be fabricated in accordance with the physical and structural requirements dictated by the use for which they are intended. A non-combustible insulating material such as diatomaceous silica, synthetic aerated silica, rock wool, magnesia, or fiberglass, properly cleaned to remove all grease and dirt, shall be used wherever insulation is necessary or desirable. Pressure relief devices (valves and/or rupture discs) must be provided to protect all compartments from overpressure failure.

The general conditions applicable to tanks are also applicable to pipes and fittings.

3.4 Transfer Procedures - Prior to transferring liquid oxygen from one container to another, all hose adapters, couplings, transfer lines and accompanying equipment shall be inspected for foreign particles. When there is a suspicion of hydrocarbons in any form, or when foreign particles are present in above equipment, the equipment shall be cleaned and inspected as indicated in Section 3.5.

After inspecting the area to determine if it is safe to commence transfer operations, hose fittings are connected to the respective container counterparts and checked for proper seating and tightness. Drip pans shall be placed under vents and connections of liquid oxygen vessels to contain spills. Liquid oxygen must not be spilled on asphalt pavement. Containers must not vent in the vicinity of combustible material.

When the transfer operation has been completed, the liquid valves on both vessels are closed and the transfer line is vented by opening an appropriate relief valve. The hose is then disconnected and allowed to warm up and preferably dry out before it is used again. Care should be taken that the hose and other accessories do not touch the ground.

Dust caps are to be replaced and exposed sections of other connectors covered, insuring that dirt, moisture, and other foreign matter cannot get into the hose and ultimately into the liquid oxygen.

3.5 Cleaning Procedures

3.5.1 General - The nature of liquid oxygen, a strong oxidizer which vigorously supports combustion, presents several unique problems when cleaning lines and storage tanks through which it passes.

Cleanliness, in the usual sense, is not a sufficient criterion when dealing with liquid oxygen systems. The high purity required prohibits the presence of solid particles of specified micron size, in addition to limiting the quantity of organic material, due to the highly reactive nature of such material in contact with liquid oxygen.

Cloth or brushes, where the material may be separated from the base, shall never be used in any cleaning operation. The acceptable types of solvent for cleaning liquid oxygen systems are chlorinated hydrocarbons such as ethylene dichloride, trichlorethylene or Freon. With the exception of Freon, which is relatively non-toxic, the problem of toxicity exists with these compounds. Carbon tetrachloride is extremely toxic and shall not be used in any cleaning operation. Relative to other solvents, operators shall not be exposed to them for long periods and safety measures must be taken for protection against vapors. When a detergent, such as a solution of tri-sodium phosphate, is used to clean liquid oxygen systems, the problem of toxicity does not exist. (NOTE: Tri-sodium phosphate is not to be used on aluminum or its alloys.)

Consideration shall be given to detached parts of liquid oxygen systems to prevent contamination of the parts or of the system from which they were removed. The best method is to encase the opening in a securely and properly applied polyethylene bag. In the absence of such a bag, wide plastic tape may be applied. Material should never be stuffed in a liquid oxygen system opening. Make-shift methods must not be used in these operations.

Since it is not possible to predict the hydrocarbon build-up rate in most liquid oxygen systems, and since it is necessary to hold extremely low hydrocarbon concentration levels in these systems, periodic inspection for contamination will determine when cleaning again becomes necessary.

3.5.2 Methods - As substitution for a lengthy detailed résumé of cleaning methods and inspection techniques for oxygen systems - presentation of which will not achieve universal acceptance - segments of two books referenced in the General Bibliography section of this document are suggested for further reading. These are:

(a) APPLIED CRYOGENIC ENGINEERING - Vance, R. W. and Duke, W. M. (Ed.) - Appendix C - Contamination Control in Cryogenic Fluids and Systems.

(b) GROUND SUPPORT SYSTEMS FOR MISSILES AND SPACE VEHICLES - Brown, K. and Weiser, P. (Ed.) - Chapter 11 - Cryogenic Missile System Hazards.

Quoting from the first of these: "There has been and still is a great variance throughout industry in the type and

size of equipment requirements, cleaning techniques, work flow, inspection methods, definition of a clean atmosphere and contamination criteria. This lack of standardization initially imposed a severe hardship on the ballistic missile effort, but standardized specifications have now been established which should reduce confusion, inefficiency, and costly recleaning operations. AIR FORCE TECHNICAL ORDER, T.O. 42C-1-11, Cleaning and Inspection Procedures for Rocket Propellant Systems (Liquid and Gaseous), governs the cleaning methods. Copies can be obtained by writing to Olmstead Air Force Base, MAOQ."

From the second: "As a result (of the extreme cleanliness requirements in the missile industry) many cleaning specifications and inspection procedures are in existence, most of them similar, but still of sufficient difference as to create confusion in the installation and acceptance of missile propellant loading systems.

"To minimize this confusion the Aerospace Industries Association, at the instigation of many of the prime contrac-

tors, sponsored a meeting . . . in an effort to formulate one set of standards which could be applicable to all missile programs. After much planning and coordinating, the AIA issued their recommendations for cleaning and inspection procedures in a pamphlet: Handbook for Contamination Control of Liquid Rocket Propulsion Systems, Aerospace Industries Association, March 7, 1960."

"In the meantime the Air Force Ballistic Missile Division issued another set of instructions governing component cleaning methods and criteria for the initial acceptance of ground support systems: Specification for Cleaning Components of Liquid Oxygen-Hydrocarbon Fuel Propellant Systems, AFBMD, ARDC, July 5, 1960."

From this it is evident that one completely acceptable set of standards does not yet exist and therefore cannot be reported here. It is recommended again, however, that the references cited at the beginning of this subsection be reviewed for extensive discussion of the topic (including cleaning and inspection procedures and techniques).

BIBLIOGRAPHY

QUALITY CONTROL OF OXYGEN PROPELLANT LIQUID OXYGEN, AVIATOR'S LIQUID BREATHING OXYGEN, AND AVIATOR'S GASEOUS BREATHING OXYGEN - Air Force Tech. Order 42B6-1-1 (May 15, 1963).

The purpose of this technical manual is to provide information, guidance, instructions, and procedures for on-base quality control of liquid oxygen used as a missile propellant, and liquid and gaseous oxygen used for aviator's breathing purposes. Included in the contents are sections on hazards and safety precautions in handling liquid and gaseous oxygen.

COMPATIBILITY OF METALLIC MATERIALS WITH LIQUID OXYGEN - Aerojet-General Corp., Rept. No. DVR 64-459 (Oct. 1964), 15 pp 4 tab 27 ref - DDC AD 459 269.

Investigation of the reactivity of metals with both liquid and gaseous oxygen were reviewed and summarized, as were theories of the ignition and propagation of combustion of metals in oxygen. Aluminum, stainless steels and nickel-based alloys were found to be compatible with liquid oxygen, but magnesium and titanium alloys were not.

OPTIMUM DESIGN OF LIQUID OXYGEN CONTAINERS - Arnett, R. W., Warren, K. A. and Mullen, L. O. - Natl. Bur. Standards, WADC TR 59-62 (Aug. 1961) Contr. AF 33(616)56-15, 235 pp 87 fig 14 tab 73 ref.

The basic parameters influencing the design of liquid oxygen containers are considered and their design interrelation evaluated. Factors considered include materials, configuration, insulation, support members, instrumentation, valves, piping, weight, evaporation loss, and accessory items such as vacuum pumps and transfer hoses. Means for evaluating and optimizing the combination of the various factors are presented together with experimental work conducted in areas where information was lacking. Description of the design and construction of a liquid oxygen

container together with the thermal test results on the container is included.

CONTAMINANTS IN LIQUID OXYGEN AS RELATED TO SAFETY IN LIQUID OXYGEN PRODUCTION AND DISTRIBUTION EQUIPMENT - Arrick, C. D. - *Advances in Cryogenic Engineering* 3, 218-25 (Proc. of 1957 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) Paper D-8, 1 fig 3 tab.

If suitable precautions are taken in production and distribution there is very little hazard from the traces of combustible contaminants present in liquid oxygen. However, these contaminants could contribute to serious fires or explosions if they are allowed to increase to dangerous levels. Combustible materials may get into liquid oxygen from the atmospheric air processed, from oil lubricated air compressor, from improperly cleaned equipment, and from equipment parts such as gaskets, packings, etc. This discussion is limited to the contaminants introduced from the air processed in a low temperature air separation plant. The bulk of the combustible materials in the liquid oxygen product usually comes from this source if reasonable attention has been paid to the other sources. It is quite practical and desirable to keep the concentration of combustibles in liquid oxygen far below dangerous levels. It is difficult and unnecessary to eliminate them completely.

STUDY OF LIQUID OXYGEN CONTAMINATION - Bailey, B. M., Sterner, C. J. and Vignale, V. J. - Air Products, Inc., Allentown, Pa., Summary Progr. Rept. No. 4 (July 1960) Contr. AF 33(616)6730, 107 pp 25 fig 12 tab 56 ref. DDC AD 253 231.

The significance of contamination in liquid oxygen is discussed. The three types of contaminants of concern in this program include: (1) combustible compounds, solid or

dissolved, which may constitute a fire or explosion hazard to both general safety and equipment; (2) solid inert contaminants which may contribute to mechanical malfunction of the propellant loading system or the rocket engine; and (3) dissolved inert contaminants which may affect the rocket thrust or, under certain circumstances, might interfere with engine ignition. The major sources of contamination consist of the air stream to the separation plant which produces the liquid oxygen, transfer of liquid oxygen from the plant to storage, and nitrogen pressurization. Nominally minor sources include vent lines and relief valves, residual contamination, and equipment deterioration. Current specifications are given for liquid oxygen and equipment.

CONTAMINATION CONTROL IN LIQUID OXYGEN SYSTEMS - Ball, W. L. - Air Products & Chemicals, Inc.

CONTAMINATION IN THE PRODUCTION & HANDLING OF CRYOGENIC FLUIDS - Smith, C. P. - Linde Company - Jour. Am. Assoc. Contamination Control 3 (Aug. 1964) pp 10-15, 19, 32. [For both papers listed above].

SAFETY IN AIR AND AMMONIA PLANTS - CEP Technical Manuals (Vol. 1-6), published by A. I. Ch. E.

Includes both published and previously unpublished data on the topic of safety in air and ammonia plants - primarily discussions and resumés of past plant operating experiences and current practices.

CORROSION EFFECTS OF LIQUID FLUORINE AND LIQUID OXYGEN ON MATERIALS OF CONSTRUCTION - Fink, F. W. and White, E. L. - Corrosion 17, 58t-60t (1961) 2 fig 1 tab 9 ref.

The corrosion behavior of materials of construction for handling liquid fluorine and liquid oxygen is summarized. This is an important matter in rocket construction. Even though both of these elements are very reactive, most of the common metals are sufficiently resistant for many applications. The compatibility with these oxidizers of alloys of iron, nickel, copper, aluminum, magnesium, titanium, and zirconium is discussed. Corrosion rate data compiled from both published and unpublished sources are presented. The compatibility of non-metals and organic materials is also reviewed. Attention is given to the effect of initiating rapid reactions, or burning of both metals and organic materials by compressive impact, tensile impact, friction, wear, and other mechanisms.

STUDY OF LIQUID OXYGEN CONTAMINATION - FINAL REPORT - Foster, R. H. - Air Products & Chem., Inc., Allentown, Pa., Final Rept. SSD-TD 62-8 (May 1961) Contr. AF 33(616)6730, 144 pp 31 fig 14 tab 62 ref DDC AD 272 377.

The purpose of this study was to develop a better understanding of the physical, chemical and mechanical relationships involved in developing realistic parameters for specification purposes for the application of oxygen to mis-

siles. The significant sources and degree of contamination are supplied as a background survey and the current specifications for liquid oxygen and ground support equipment are discussed. Recommendations for liquid oxygen specification and for equipment operation are presented. Sources and mechanisms for ignition of liquid oxygen systems, factors related to solid contaminants, cleaning and purification of oxygen equipment and handling systems, have been included. Also as part of this contract, the contractor developed safety standards for use in high pressure oxygen and helium gases for later incorporation in the Liquid Propellant Safety Manual published by the Liquid Propellant Information Agency. As part of this program, a three-month analytical survey was made at Cape Canaveral and summarized herein.

FIELD HANDLING OF LIQUID BREATHING OXYGEN - Frederick Research Corporation - NAVWEPS 06-30-501 (Aug. 1959) 76 pp 68 fig 4 tab 40 ref.

Personnel responsible for the servicing of aircraft liquid oxygen converter systems should be thoroughly familiar with the physical and chemical characteristics of oxygen and with the pertinent operational instructions. The purpose of this handbook is to provide field personnel with proper instructions for the safe and efficient handling of liquid oxygen, and to familiarize all interested personnel with the scope of the liquid oxygen ground support system.

IGNITION IN HIGH PRESSURE OXYGEN - Guter, M. British Oxygen Company R&D Rept. 1312 - (First issued June 1950, Ministry of Supply; Re-issued Feb. 1967, Ministry of Aviation) - DDC AD 648 612.

The objective of this work was to determine the ignition temperatures of selected materials in oxygen at pressures up to 250 atmospheres, and to study the influence of a number of variables on this behaviour. In addition to pressure, the main variables examined include rate of flow of gas, rate of heating the sample, effect of prolonged storage in oxygen under pressure, oxygen concentration and the physical state of the sample. No attempt was made to study the behaviour of materials under actual working conditions, or to decide if they would be completely safe against all hazards in use. This could only be done by means of a greatly enlarged program of research relating to the design and use of particular items of equipment.

In view of the very large number of materials that have been examined, a broad classification has been attempted and the materials have been placed in the following five groups:

- I Lubricants, including thread sealing compounds.
- II Natural and synthetic rubber hose materials.
- III Polymers.
- IV Valve seat materials.
- V Metals and alloys.

The report first describes the apparatus and experimental methods used. The results are presented and discussed in

five separate sections relating respectively to the above groups.

Results of other workers in this field are few and have been found only for materials classified in sections I, IV and V. These results and an account of the methods by which they were obtained are included in the relevant discussion sections.

MECHANICALLY INITIATED REACTIONS OF ORGANIC MATERIALS IN MISSILE OXIDIZERS - Hauser, R. L., Sykes, G. E. and Rumpel, W. F. - Martin Co., Denver, Colo., ASD Tech. Rept. 61-324 (June 1960 - June 1961) Contr. AF 33(616)7271, 281 pp 50 fig 137 tab 8 ref.

This report presents the results of impact testing of 24 organic materials with liquid oxygen. In addition, nine of these materials were tested with nitrogen tetroxide. Pure polymers, plasticizers, and antioxidants were studied and their threshold sensitivity levels and detonation energies were determined. Procedures and equations for calibrating impact testing machines were developed and used to calculate the rates of energy transfer into test materials. A full record of test procedures is included. In addition, 18 of the given materials in contact with liquid oxygen were subjected to shear forces with a modified Shell Four-Ball Wear Tester to determine whether reactions could be initiated by friction.

REACTIONS OF ORGANIC MATERIALS WITH LIQUID OXYGEN - Hauser, R. L. and Rumpel, W. F. - Advances in Cryogenic Engineering 8, 242-50 (Proc. of 1962 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1963) Paper B-2, 5 fig 2 tab 4 ref.

This paper summarizes the pertinent results of a scientific study of the nature and magnitude of detonation reactions involving organic materials and liquid oxygen. The study is more complete than any available in current literature. Theoretical analysis and instrumentation techniques now permit calibration of impact testing machines and calculation of energy transfer rates. A number of pure polymers, particularly elastomers, were found to be less reactive than their commercial counterparts; some plasticizers and antioxidants were observed to propagate in liquid oxygen, and none was initiated in gaseous oxygen.

Relative measures of detonation energies were obtained for a number of organic materials; this information provides a safer basis for selection of materials than has been available solely from reaction frequency data.

CORROSION IN CRYOGENIC LIQUIDS - Jackson, J. D. - Chem. Eng. Progress 57, 61-64 (1961) 6 fig 10 ref.

The large use of liquid propellants in missile systems has brought many serious problems to the missile designer and the materials engineer. One important problem is the corrosion behavior of materials of construction under the various exposure conditions of the missile and the auxiliary equipment. In the missile, short-term exposure occurs; however, the materials may be stressed almost to their yield strength. In the auxiliary equipment (such as storage tanks, pipelines, and pumps), long term exposure, under

much less severe strength requirements, occurs. This article discusses corrosion behavior and mechanical properties of metals used in handling liquid oxygen and liquid fluorine.

REACTIVITY OF METALS WITH LIQUID AND GASEOUS OXYGEN - Jackson, J. D., Boyd, W. K. and Miller, P. D. Battelle Memorial Inst., Defense Metals Information Center, Columbus, Ohio, DMIC Memo No. 163 (Jan. 1963) 26 pp DDC AD 297 124.

Since the first observation of a violent reaction in early 1959, the compatibility of titanium and its alloys with liquid oxygen (LOX) has received considerable attention. Initially, laboratory investigations were primarily limited to impact studies utilizing the ABMA impact tester or modifications thereof. Later the Air Force initiated a program to determine the mechanism of the reaction. The results of these early studies were previously summarized in DMIC Memorandum 89, dated March 8, 1961.

More recently, the factors necessary to promote reactions between titanium and liquid or gaseous oxygen have been studied under conditions similar to those which would be encountered in missile and space service. It is the purpose of this memorandum to summarize the present state of the art in the light of both past and present developments.

HAZARD LEVEL OF HYDROCARBON FILMS IN SYSTEMS CONTAINING LIQUID AND GASEOUS OXYGEN - Kehat, E. - Advances in Cryogenic Engineering 7, 163-69 (Proc. of 1961 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1962) Paper B-4, 8 fig 1 tab 8 ref.

The object of this work was to determine realistic standards of cleanliness in systems containing liquid and gaseous oxygen. An arbitrary figure of 4 mg/ft² had been set by examination of systems that had caused no trouble in the past. It was felt that this figure was too low and that experimental determination of the safe level should be made to set such standards.

EFFECT OF LIQUID NITROGEN DILUTION ON LOX IMPACT SENSITIVITY - Key, C. F., Gayle, J. B. - NASA-George C. Marshall Space Flight Center, Huntsville, Alabama - NASA TMX-53208 (Feb. 15, 1965). [See J. Spacecraft Rockets 3, n. 2, 274-76 (Feb. 1966) for summary publication.]

An experimental investigation was carried out to study the decrease in reactivity of materials with liquid oxygen (LOX) that is caused by dilution of the LOX with liquid nitrogen (LN₂). A wide range of materials was selected for testing, each of which previously had been shown to be sensitive to impact in LOX. Tests were made with the

ABMA LOX Impact Tester using LOX/LN₂ mixtures ranging in concentration from 20 percent LOX in LN₂ to pure LOX. The results showed that relatively large proportions of LN₂ were required to effect an appreciable decrease in reactivity; however, all materials tested were insensitive to impact at 10 Kg-m in liquid air.

COMPATIBILITY OF MATERIALS WITH LIQUID OXYGEN - Key, C. F. and Riehl, W. A. - NASA - George C. Marshall Space Flight Center, Huntsville, Ala., Internal Report MTP-P&VE-M-63-14 (Dec 4, 1963).

The test instrument and procedure developed by Lucas and Riehl was used to determine the compatibility of a wide variety of materials with liquid oxygen (LOX). This method is based upon the tendency of materials to react with LOX on impact and is commonly known as the "ABMA Tester". Within the past eight years' use, over 100,000 individual test drops have been made on approximately 1,000 different materials.

Pertinent data from these tests have been compiled and the findings are presented in this report. Recommendations are made for guidance of designers and others in selection of safe materials for use in oxygen systems. Materials are discussed according to the following classifications: (1) Lubricants, (2) Sealants and Threading compounds, (3) Thermal and Electrical Insulation, (4) Elastomers, Plastics and Adhesives, (5) Gaskets and Packing, (6) Metals, Alloys and Solders, (7) Dye Penetrants, and (8) Solvents, Cleaning Solutions and Miscellaneous.

COMPATIBILITY OF MATERIALS WITH 7500 PSI OXYGEN - Nihart, G. J. and Smith, C. P. - Linde Company - AMRL-TDR-64-76 (Oct. 1964) Contr. AF 33(657)-11686, 89 pp 43 fig 12 tab 17 ref. - DDC AD 608 260.

A research program was conducted to develop ignition data on thread lubricants, thread sealants, fluorocarbon plastics, and metals. Spontaneous ignition temperatures were determined in both 2000 psi and 7500 psi oxygen for all the above materials except metals. The spontaneous ignition temperatures for these materials were found to be essentially the same in 7500 psi oxygen and in 2000 psi oxygen. Only three of the tested lubricants are recommended for possible use in 7500 psi systems. None of the thread sealants are recommended. Glass-filled polytetrafluoroethylene is usable only if tightly confined. The relative ease of ignition of metals and alloys was determined by promoted ignition methods in oxygen at 7500 psi. Inconel alloy 600, brass, Monel alloy 400, and nickel were found to have the highest resistance to ignition and combustion among the common alloys and metals. Of the materials tested, stainless steel and aluminum are the least satisfactory for use at oxygen pressures of 7500 psi. A test system was constructed to evaluate the hazards in rapidly charging a 65 cubic inch nickel-lined vessel with high pressure oxygen. A series of rapid charging tests up to as high as 8000 psi proceeded without incident. Electrostatic charges measured during the charging were negligible.

PRECAUTIONS AND SAFE PRACTICES FOR HANDLING LIQUEFIED ATMOSPHERE GASES - Linde Company, Div. of Union Carbide Corp., Publication F-9888.

The purpose of this booklet is to outline the basic techniques for the safe handling of liquefied atmospheric gases.

LONG-TERM STORAGE OF LIQUID OXYGEN - Little, Arthur D., Inc. (July 1958) Contr. AF 04(647)-130, 24 pp

4 fig 5 tab 2 ref - DDC AD 267 782.

This report covers an investigation of the hazards produced by the long-term storage of liquid oxygen. Possible contamination in the transfer process from the plant to the storage tank was not of concern here, but only those hydrocarbons occurring in the liquid oxygen received from the air-separation plant.

SURVEY OF HAZARDS OF HANDLING LIQUID OXYGEN - McCamy, C. S. - Ind. Eng. Chem. 49, No. 9, 81A-82A (Sept. 1957) 15 ref.

With the increased industrial use of liquid oxygen, the importance of knowing about its compatibility with other materials is very much accentuated. Included in this article are properties of liquid and gaseous oxygen, materials compatibility with oxygen, flammability and ignition characteristics, etc.

OXYGEN PLANT SAFETY PRINCIPLES - McKinley, C. and Himmelberger, F. - Chem. Eng. Progr. 53, No. 3, 112-21 (March 1957) 6 fig 6 tab.

Safe operation of air separation equipment is a subject of growing importance because of the vastly increased rates of tonnage oxygen and nitrogen usage by the chemical and metallurgical industries. Much of the large new demand is at locations under heavy and increasing air pollution - conditions requiring fullest application of present technology. Reduction of hazards requires understanding. To this end, past published data provides limited knowledge.

Original data developed at Air Products by controlled explosion tests and solubility studies upon combustible contaminants permit the formulation of new safety principles consistent with historical findings.

This article presents new information on the character of materials and explosions in oxygen plants, and is not intended as a review of design and operating practice.

STANDARD FOR BULK OXYGEN SYSTEMS AT CONSUMER SITES - National Fire Protection Association, Rept. No. 566 (May 1962) 8 pp.

These Standards cover the general principles recommended for the installation of bulk oxygen systems on industrial and institutional consumer premises. It covers requirements for bulk oxygen systems including design, location, operation and maintenance. The Standards do not apply to oxygen manufacturing plants or other establishments operated by the oxygen supplier or his agent for the purpose of storing oxygen and refilling portable containers, trailers, mobile supply trucks or tank cars.

COMPATIBILITY OF MATERIALS WITH LIQUID OXYGEN - Peckham, H. M. and Hauser, R. L. - Advances in Cryogenic Engineering 4, 26-46 (Proc. of 1958 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) Paper A-3, 6 fig 1 tab.

Insuring the compatibility of materials with liquid oxygen has been a problem with the missile industry from the first use of LOX in propellant systems. Many materials are compatible under static conditions; however, when they are used in missile systems where dynamic or shock produc-

ing conditions are ever present, a severe hazard of detonation exists. The cryogenic industry has been aware of this problem for some time, although the study to date has been restricted to solving the immediate problems of producing and transporting the fluid.

The first active study of the compatibility problem in the missile industry was focused on lubricants. Investigations began in early 1957 when it was found that most commercial lubricants were impact sensitive in combination with LOX.

This paper reviews the application of (compatibility tester) standards to a test apparatus constructed for the Materials Engineering Laboratory at Martin-Denver and presents results of (materials compatibility) tests conducted by Martin-Denver and others.

SAFETY ASPECTS IN THE DESIGN AND OPERATION OF OXYGEN SYSTEMS - Reynales, G. H. - Douglas Aircraft Co., Eng. Paper No. 713 (Jan 1959).

COMPATIBILITY OF MATERIALS WITH OXYGEN - Reynales, G. H. - Douglas Aircraft Co., Rept. D81-444 (Oct. 1958) 78 pp 26 ref.

The contents of this report represent the result of a rapid survey made to meet an immediate need for general information on the behavior of oxygen in conjunction with the materials used in the construction of WS-315A, ground support equipment. The study covers the review of technical literature, test reports and unpublished data concerning the behavior of gaseous and liquid oxygen.

SELECTION OF LUBRICANTS AND THREAD COMPOUNDS FOR OXYGEN MISSILE SYSTEMS - Reynales, G. H. - *Advances in Cryogenic Engineering* 6, 117-29 (Proc. of 1960 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1961) Paper B-6, 34 fig 7 tab.

The missile industry has been concerned from the beginning about the presence of lubricants in oxygen systems because the use of some of these compounds may lead to burnouts and to disastrous explosions. This brief study aims to analyze the reasons behind the special role played by these compounds in missile oxygen systems and to find means of reducing the hazards resulting from their usage by evolving criteria for selecting safe compounds.

REACTIVITY OF TITANIUM WITH OXYGEN - Riehl, W. A., Key, C. F. and Gayle, J. B. - NASA - George C. Marshall Space Flight Center, Huntsville, Ala. Internal Report MTP-P&VB-M-62-13 (Nov. 30, 1962).

The reactivity of titanium with oxygen was studied by several test methods and under a variety of conditions associated with space vehicles.

Titanium is highly sensitive to impact in contact with LOX, and this method was used to study the effects of surface treatments, coatings, and numerous other factors upon the reactivity. The comparative reactivities of titanium, aluminum, and stainless steel alloys with oxygen were in-

vestigated by impact, shock, puncture, and spark sensitivity testing. Punctures resulting from bullets, darts, pins, or artificial meteoroids usually caused explosions. Coatings which reduced titanium reactivity in impact or shock tests were not beneficial under puncture conditions. Aluminum and stainless steel failed to react on impact or puncture.

The shock stimuli produced by small detonator caps alone were sufficient to initiate explosive reaction of titanium in contact with oxygen. An extremely heavy shock was necessary to cause aluminum to react under the same test conditions, and stainless steel did not react under the most drastic shock conditions employed. The titanium/oxygen combination is considerably more susceptible to spark initiation than aluminum/oxygen. A comparatively high energy spark was necessary for reaction of 0.010-inch-thick sheets of titanium with oxygen.

Under the particular test conditions used, titanium was insensitive to reaction with oxygen when subjected to vibration, pressure cycling, or to rupture with pressurization of thin-walled tanks containing LOX.

IGNITION OF METALS IN OXYGEN - White, E. L. and Ward, J. J. - Battelle Mem. Inst., Defense Metals Inform. Center, Columbus, Ohio - DMIC Memo. No. 224 (Feb. 1, 1966) 55 pp 12 fig 13 tab 46 ref.

The report deals with the ignition of metals in oxygen and oxygen-containing atmospheres. The ignition of metals is reviewed from the viewpoints of (a) methods that have been used to study behavior, (b) experimental values that have been obtained, and (c) the status of theories that permit the calculations of ignition temperatures. A number of experimental methods have been used to determine the ignition temperature of solid metals and alloys in oxygen gas, air, various mixtures of inert gases with oxygen, and liquid oxygen. In addition to the input of energy from heat sources, the effects of electrical-energy input, and various types of mechanical-energy input on the ignition temperature have also been reported. Experimental values of ignition temperature are discussed for alloys of titanium, aluminum, copper, nickel, iron, cobalt, magnesium, tin, and lead; stainless steels, silver and silver solders, and other metals and alloys are also discussed.

SAFETY IN THE USE OF OXYGEN - Voit, R. - Linde Ber. Tech. u. Wiss. 9, 46-55 (Sept. 1960) 10 fig.

This paper discusses the three following tasks undertaken by the author's company:

1. The investigation of the dangers concomitant with the liquefaction of air and the fractionation into its constituent parts.
2. Determination of the causes of burns and injuries to attendants in oxygen plants, - there being no simultaneous injury to the plant - and the elimination of these accident possibilities.
3. Investigation of the causes of fires and explosions at oxygen valves, lines and plants, and their elimination.

SAFETY ENGINEERING AS APPLIED TO THE HANDLING
OF LIQUEFIED ATMOSPHERIC GASES - Zenner, G. H. -
Advances in Cryogenic Engineering 1, 291-95 (Proc. of
1954 Cryogenic Eng. Conf.) Plenum Press, Inc., New York
(1960) Paper H-5, 4 fig.

In the development of the present large-scale production
and distribution of liquefied atmospheric gases many unusu-
al hazards were encountered. The following discussion is
intended to clarify these hazards and to outline some of the
means used to overcome them.

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PHYSICAL PROPERTIES OF LIQUID OXYGEN

PROPERTY VALUES OF OXYGEN
AT SELECTED CONDITIONS

Nomenclature and Conditions

TP = Triple Point

NBT = Normal Boiling Temperature (and 1 atm.)

NTP = Normal Temperature and Pressure
(70°F, 14.7 psia)

273.15°K = 0°C = 32°F = 491.67°R

The term "mole" as used here means "gm-mole."

a. Calculated from density of the liquid and dP/dT of fusion line by the Clapeyron equation.

*Changes have been made to correct data to this scale where necessary.

Property Values of Oxygen at Selected Conditions						
Property			Value	Reference		
Molecular Weight			31.9988	1		
Triple Point Values	Temperature, °K		54.353	2, 3		
	Pressure, mm Hg		1.14	2, 3		
	Density, mole/cc	Solid	0.0430	a		
		Liquid	0.0413	4, 5		
		Vapor	0.000000336	4, 5		
Normal Boiling Values	Temperature (T _b), °K		90.180	2, 3		
	Density, mole/cc	Liquid	0.03565	5, 6, 22		
		Vapor	0.0001396	5, 22		
Critical Values	Temperature, °K		154.77	2, 3		
	Pressure, mm Hg		38109	2, 3		
	Density, mole/cc		0.013333	3, 22		
One Liter Liquid (NBT) Equivalents	Weight, kg		1.141	6, 22		
	Volume of Gas, liters	NBT	255.4	6, 22		
		NTP	860.1	4, 6, 2		
		Equivalent Volumes of Gas per Volume of Liquid (NBT)		NBT	255.4	6, 22
		NTP	860.1	4, 6, 2		
Heat of Fusion, Cal/mole			TP	106.3	4, 7, 8	
Heat of Vaporization, Cal/mole			NBT	1630.9	5, 7, 22	
Specific Heat Cal/mole-°K	C _s	Solid	TP	11.1	5, 8	
		Liquid	NBT	13.0	5, 8	
		Vapor	NBT		c	
	C _v	Liquid	NBT	7.7	d	9
		Vapor	NBT	5.0	d	10, 11
		Gas	NTP	5.03		2
	C _p	Liquid	NBT	13.0		6, 8
		Vapor	NBT	7.1	d	11
		Gas	NTP	7.03		2
Specific Heat Ratio	C _p /C _v	Liquid	NBT	1.69	6, 8, 9	
		Vapor	NBT	1.42	10, 11	
		Gas	NTP	1.40	2	
Thermal Conductivity K Cal/cm-sec-°K	Liquid	NBT	0.0003575	12, 13		
	Vapor	NBT	0.00001930	14		
	Gas	NTP	0.0000626	15, 14, 2		
Viscosity μ Gm/cm-sec	Liquid	NBT	0.00190	16, 17, 21		
	Vapor	NBT	0.0000692	18, 19, 13		
	Gas	NTP	0.000203	15, 13, 2, 20		

b. The Advisory Committee on Thermometry of the International Committee on Weights and Measures has agreed on 90.17°K as the present most probable value of thermodynamic temperature for the normal boiling point of oxygen; see Brickwedde, F. G., "International Practical Temperature Scale," Physics Today 16, 24-26 (1963).

c. C_p (vapor)_{NBT} could be used here.

d. Value taken at 90°K (-297.67°R).

Property Values of Oxygen at Selected Conditions				
Property			Value	Reference
Molecular Weight			31.9988	1
Triple Point Values	Temperature, °F		-361.83	2, 3
	Pressure, psia		0.0220	2, 3
	Density, lb/ft ³	Solid	85.9	a
		Liquid	82.5	4, 5
Vapor		0.000671	4, 5	
Normal Boiling Values	Temperature (T _b), °F		-297.35	2, 3
	Density, lb/ft ³	Liquid	71.2	5, 6, 22
		Vapor	0.2789	5, 22
Critical Values	Temperature, °F		-181.08	2, 3
	Pressure, psia		736.90	2, 3
	Density, lb/ft ³		26.63	3, 22
One Gallon Liquid (NBT) Equivalents	Weight, lb		9.520	6, 22
	Volume of gas, ft ³	NBT	34.15	6, 22
		NTP	115.1	4, 6, 2
		Equivalent Volumes of Gas per Volume of Liquid (NBT)		NBT 255.4
		NTP 860.1	4, 6, 2	
Heat of Fusion, Btu/lb			TP 5.976	4, 7, 8
Heat of Vaporization, Btu/lb			NBT 91.738	5, 7, 22
Specific Heat Btu/lb-°F	C _s	Solid TP	0.346	5, 8
		Liquid NBT	0.405	5, 8
		Vapor NBT		c
	C _v	Liquid NBT	0.24	d 9
		Vapor NBT	0.16	d 10, 11
		Gas NTP	0.157	2
	C _p	Liquid NBT	0.406	6, 8
		Vapor NBT	0.22	d 11
		Gas NTP	0.220	2
Specific Heat Ratio	C _p / C _v	Liquid NBT	1.69	6, 8, 9
		Vapor NBT	1.42	10, 11
		Gas NTP	1.40	2
Thermal Conductivity K Btu/hr-ft-°F		Liquid NBT	0.08643	12, 13
		Vapor NBT	0.004666	14
		Gas NTP	0.01515	15, 14, 2
Viscosity μ Centipoise		Liquid NBT	0.190	16, 17, 21
		Vapor NBT	0.00692	18, 19, 13
		Gas NTP	0.0203	15, 13, 2, 20

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III. RECOMMENDED MATERIALS AND PRACTICES FOR USE WITH LIQUID HYDROGEN

- A Compilation from the Literature with Annotated Bibliography -

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BIBLIOGRAPHY

1. HAZARDS

1.1 Health - The health hazards of liquid hydrogen arise from its very low temperature and the fact that the gas can exclude oxygen, thus causing asphyxiation. If liquid, or cold gaseous, hydrogen contacts the skin, damage resembling burns can result. The extent of such damage can range from relatively minor burns to complete embrittlement and destruction of exposed tissue. The immediate effects of freezing by liquid hydrogen can be minimized by soaking affected parts in tepid water. Extensive burns require prompt medical attention.

1.2 Fire.

1.2.1 General - An unconfined mixture of hydrogen and air will burn but not detonate if it is exposed to a limited ignition source such as a spark. In confined areas or when ignition is accomplished by a shock source, equivalent to a blasting cap or a small explosive charge, a detonation, or an explosion of the mixture can occur. A hydrogen flame has one-tenth the radiation effect and one-tenth the duration of hydrocarbon fuels such as gasoline and kerosene. When no impurities are present, hydrogen burns in air with an invisible flame. Hydrogen-air mixtures containing as little as 4 percent or as much as 74 percent hydrogen by volume are readily ignited. Hydrogen-oxygen mixtures are flammable over the range of 4 to 94 percent hydrogen by volume.

Quantity-distance relationships for the storage and use of hydrogen have been prepared and published by the U.S. Dept. of Defense, Air Force, Navy, Compressed Gas Association, Bureau of Mines, National Fire Protection Association, U.S. Air Force and others; however, to date, general acceptance for any one of these guides has not been achieved. Each requirement, therefore, should be examined and evaluated with current information from the above sources in mind, and with the understanding that only the best engineering judgment is good enough to make necessary decisions in this matter.

1.2.2 Control - The most effective control of hydrogen fires is the shutting off of the supply. Equipment should be designed for effective control and isolation in case of failures. Fires from hydrogen gas can be controlled effectively with the common extinguishing agents such as water, carbon dioxide and steam. It should be remembered, however, that if hydrogen flame resulting from leaks are extinguished, hydrogen will continue to leak and form a cloud of combustible gas which may explode if ignited. Where large spills occur, vacate the area at least 400 ft radius from the source. It should be emphasized that the outer limits of the flame, or fire, cannot generally be seen. If leaks occur in enclosed areas, care should be exercised to eliminate ignition possibilities, and adequately ventilate before entering.

1.2.3 Prevention - The following sources of ignition must be eliminated to prevent the occurrence of fires:

1. Open Flames - smoking, welding, or open flame shall be prohibited when hydrogen is being processed.

2. Electrical Equipment - all electrical equipment must be of the explosion-proof type or must be purged continuously with an inert gas.

3. Metallic Sparks - all handling tools should be of the spark resistant, non-ferrous type or should be covered with a non-conducting material.

4. Static Electricity - all equipment must be properly grounded, and the use of conductive floors and shoes is recommended.

1.3 Explosion.

1.3.1 General - Liquid hydrogen is not in itself explosive but if contaminated with oxygen it is unstable and an explosion is likely to occur. The liquid is hazardous, however, because the gas is always present due to evaporation. An explosive hazard exists when the hydrogen-air mixture is completely or partially confined. Such a mixture will propagate a detonation wave when initiated by an explosive. A deflagration will occur when this mixture is ignited from a spark source. However, either type of ignition will cause serious damage. Explosive hazards also exist when oxygen enriched solid air or when strong oxidizers are present. Pressure rupture, with severe consequences, can occur when the liquid is held in a closed system with no refrigeration. Hydrogen cannot be maintained as a liquid if its temperature rises above the critical temperature (-400 F) regardless of confining pressure. Liquid hydrogen trapped between valves can cause violent rupture of the pipe while loss of refrigeration can cause a storage tank to rupture if the pressure is not relieved by suitable devices. Liquid hydrogen does not present a detonable hazard when it evaporates and mixes with air in an unconfined space, although certain mixtures are subject to rapid combustion with a very high rate of flame propagation.

1.3.2 Prevention - All sources of ignition shall be kept away from areas where liquid hydrogen is being stored or handled. This means no smoking, use of approved explosion-proof electrical equipment where available and proper grounding of equipment to remove static electricity. Venting of hydrogen vapors should be accomplished at a remote location and storage tanks and other containers should be kept under positive pressure to insure that air does not enter the system. An "Underwriter Approved" automatic device for detecting hazardous concentrations of hydrogen should be installed where appropriate. Pressure rupture of materials can be avoided by the proper use of pressure relief valves and blow-out discs. Pressure gages should also be used for system monitoring. Enclosures of any type that would allow trapping of hydrogen should be either eliminated or ventilated. Careful pressure and leak testing of all lines should occur periodically.

2. SAFETY MEASURES

2.1 General - Liquid hydrogen is difficult to handle because of its low temperature, which causes many materials

to become brittle. The low temperature also constitutes a freezing hazard to personnel who come into direct contact with the fluid or any unprotected equipment. However, the most serious hazard associated with the use of liquid hydrogen is the danger of fire or explosion. Liquid hydrogen is extremely volatile; the flammable and detonation limits of gaseous mixtures of air and hydrogen are wide; and the potential energy release per pound of reactants is very large. All operations involving the handling of liquid hydrogen shall be performed by two or more persons working in a group. Trained supervision of all potentially hazardous activities involving liquid hydrogen is essential.

2.2 Personnel Education - The following subjects shall be explained to all personnel concerned with liquid hydrogen handling, transfer, and storage:

(a) Nature and properties of hydrogen in both the liquid and the gaseous phases.

(b) Approved materials which are compatible with liquid hydrogen.

(c) Proper equipment and its operation.

(d) Use and care of protective equipment and clothing.

(e) Safety, self-aid, and first-aid instructions.

2.3 Personal Protection - The principal hazards associated with the handling of liquid hydrogen are fire and the extremely low temperature of the liquid.

For hand protection, gauntlet type gloves which can be easily and rapidly removed are satisfactory. These gloves may be either of asbestos or chrome leather with an inner liner of impermeable material. For protection of the feet, leather shoes which can be readily removed should be used. These can be high top or low top; the choice depends entirely on the area in which the individual will be working. If high top boots are used, pants legs will be outside the boot tops. Where soles of shoes have been exposed to the liquid, the footwear should be removed immediately to prevent delayed frostbite.

Head and face protection requires the use of acid-type goggles or a face shield to stop splashes. Flame-resistant and static-free clothing should be worn by personnel working with liquid hydrogen because of the fire hazard.

Respiratory protection is not required; however, the possibility of asphyxiation in closed areas should be recognized. The use of oxygen breathing equipment in a hydrogen atmosphere will create an explosion hazard and shall be avoided.

3. TRANSFER AND STORAGE

3.1 General - Liquid hydrogen must be stored in containers (either fixed or mobile) of approved design, materials, and construction.

Storage, transfer, and test areas must be kept neat and free from combustibles. These areas must be inspected frequently.

An adequate water supply or fire extinguishers must be available for combating fires (of combustibles other than hydrogen). Approved deluge-type personnel showers should

be properly located for immediate use in an emergency.

3.2 Materials - The ability of materials to maintain satisfactory physical properties and to withstand thermal stresses caused by large temperature changes is of prime importance.

3.2.1 Metals - The ferrous alloys, except the austenitic chromium-nickel alloys, lose their ductility when subjected to the low temperatures of liquid hydrogen and depending on their form and the application to which they are applied they may become too brittle for use with liquid hydrogen. Metals suitable for this service are aluminum, copper, nickel, and most of their alloys, as well as the "300 series" austenitic stainless steels.

3.2.2 Non-Metals - Several of the non-metals suitable for use with liquid hydrogen are Teflon, Kel-F, Dacron, Nylon, Mylar and Micarta. The most commonly used insulating materials for cryogenic application (with due recognition being given to the classic high-vacuum insulation) are expanded perlite, silica aerogel, diatomaceous earth, fiberglass, polystyrene and polyurethane plastic foams, cork, balsa and asbestos.

3.2.3 Hydrogen Embrittlement - Although it is not considered generally to be a cryogenic problem, the term hydrogen embrittlement describes any of several undesirable phenomena which users of hydrogen gas handling equipment must consider in any serious, comprehensive safety analysis. Such equipment may range from the small Bourdon tube in a hydrogen gas measuring pressure gage to a very large high pressure hydrogen gas storage cylinder in a multi-million standard cubic feet tank farm - either or both items possibly being used in conjunction with a liquid hydrogen production, test, or rocket launch facility.

Of the various ways in which hydrogen may embrittle metallic structures or components, the mechanism of primary importance to this discussion is the hydrogen-induced delayed brittle fracture of high strength structural steels at relatively low applied stresses - otherwise known as hydrogen-stress cracking. Using the previously mentioned items as typical examples, instances have been cited where ferritic Bourdon tubes containing hydrogen have burst at pressures as low as one-tenth full scale, whereas prior testing to the rated full scale pressure with other fluids proved uneventful; also, various high pressure (5000 psi) gas receivers failed (cracked) in hydrogen gas service well below the design pressure after initially passing a 7500 psi hydrostatic proof test, whereas high pressure nitrogen gas receivers of the same manufacture for the same pressure duty experienced no problems in this regard.

It is not the purpose of this section to present a treatise on the subject of hydrogen embrittlement. Rather, the intent is to state the problem (above), summarize pertinent observed facts concerning the matter, and suggest certain solutions. The observations and some of the more obvious solutions are:

1) Embrittlement to any appreciable depth is uncommon in the austenitic stainless steels; it is common in the ferritic steels under the right conditions.

2) Embrittlement increases with hardening or cold-

working. High strength steels are particularly susceptible (no failures having been confirmed in steels with ultimate tensile strengths below 94,000 psi).

3) This form of embrittlement only occurs within about 100 C of ambient temperature.

4) Embrittlement reduces ductility of the material, but this is recovered upon removal of the hydrogen.

5) The problem is alleviated or dismissed by substitution of non-embrittling (e.g., non-ferritic, non-cold-worked) materials for the undesirable ones, if possible, or by imposing non-permeable, barrier coatings or liners between the hydrogen and the sensitive or susceptible material.

As a final note, the present state of understanding of the several phenomena gathered under the heading of hydrogen embrittlement is incomplete. Various reports and recent reviews on the subject are available from such places as the Battelle Memorial Institute, and the reader is referred to these research and information centers for more specific knowledge.

3.3 Equipment - Liquid hydrogen handling equipment shall be degreased by washing with approved grease-removing solvents before being used. Equipment taken out of service for maintenance or modification shall be inspected and cleaned before being returned to service.

Liquid hydrogen may be stored in either fixed or mobile tanks of approved design and materials. Storage and shipping containers designed for non-cryogenic service shall not be used in this service. Storage tanks shall be proof-tested prior to service in accordance with the provisions of applicable ASME, ASTM, or ICC specifications for pressure vessels. Containers for shipment, storage, and transfer of liquid hydrogen should be fabricated in accordance with the physical and structural requirements dictated by the use for which they are intended. Pressure relief devices (valves and/or rupture discs) must be provided to protect all compartments from overpressure failure.

The general conditions applicable to tanks are also applicable to pipes and fittings.

3.4 Transfer Procedures - Prior to transferring liquid hydrogen from one container to another, all hose adapters, couplings, transfer lines and accompanying equipment shall be inspected for cleanliness. Connector O-rings shall be examined for cracks or other signs of damage and replaced when necessary; these seals should be lightly coated with silicone vacuum-grease.

After inspecting the area to determine if it is safe to commence transfer operations, hose fittings are connected to the respective container counterparts and checked for proper seating and tightness. Static grounding cables shall always be used, between both the transferring vehicles and strategically located ground rods, in transfer and storage of liquid hydrogen.

The system should now be put through a sequence of evacuation and purge cycles - initially with an inert gas such as dry nitrogen and then with hydrogen. The first cycle may also be used as a means of detecting and correcting leaks. Alternate cycles of hydrogen pressurization and vented depressurization are, upon occasion, also useful

in reducing system gas contamination to an acceptable level when evacuation is impossible or impractical. After the system has finally been charged with hydrogen, it should be maintained at slight positive pressure to prevent infiltration, or inward diffusion, of undesirable fluids (air, water, etc.).

Upon completion of the transfer, the system may be "inerted" by permitting, or causing, it to warm above the liquefaction temperature of nitrogen, followed by an adequate number of evacuation and nitrogen gas purge cycles. Again it is to be left under slight positive pressure, and consideration must be given to subsequent warming of the contained gas with resultant pressure rise.

If the lines are to be disconnected upon completion of the transfer operation, disregard the preceding paragraph. Close valves on both vessels and vent the transfer line by opening an appropriate relief valve. After disconnecting the line, dust caps are to be replaced and exposed sections of other connectors covered, insuring that dirt, moisture or other foreign matter cannot get into the hose and ultimately

into the liquid hydrogen.

Several approaches have developed on the subject of venting hydrogen gas. One constitutes deliberate burning or flaring of the escaping gas, while the other involves venting without burning. A certain degree of hazard exists in both cases, however, and the final choice rests with conditions existing at the facility - flow rates, location of vent stack, solid contaminant concentration level in vent gas, etc. If the gas is flared, provision must be made to prevent flashback down the stack; if it is not flared, reasonable assurance should exist that ignition will not occur due to electrostatic phenomena. One means of preventing a flow of air down the stack during periods of minimum, or no, venting is to provide a continuous nitrogen purge up the stack. When possible, and economically feasible, it is desirable to recover the gas, which would otherwise be discharged to atmosphere, by collecting it in gasholders and compressing it to high pressure storage.

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SAFETY ASPECTS OF LIQUID HYDROGEN - Allan, D. S. - Little, Arthur D., Inc. - Society of Automotive Engineers Paper No. 994B (Jan. 1965) 5 pp 3 fig 5 ref.

The results of both research and practical experience that have provided a working basis for the safe storage and handling of liquid hydrogen are reviewed. The hazards involved in the use of liquid hydrogen, including those related to both its cryogenic characteristics and its reactivity with air, are discussed. General recommendations are made as to precautionary measures which shall be considered in the design and operation of liquid hydrogen facilities.

TEST EQUIPMENT AND PROCEDURES USED IN THE DEVELOPMENT OF LIQUID OXYGEN - HYDROGEN ROCKET ENGINES - Anschutz, R. H. - Advances in Cryogenic Engineering 5, 62-8 (Proc. of 1959 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960).

The development of rocket engines utilizing cryogenic fluid propellants involves many specialized tests and the use of much specialized test equipment. This is particularly true where one of the propellants used is liquid hydrogen with its extremely low temperature and wide flammability limits. Pratt & Whitney Aircraft is currently developing for the Air Force under NASA direction the XLR 115 liquid oxygen-liquid hydrogen rocket engine. This paper discusses some of the test equipment and procedures which are being employed in the XLR 115 engine development program at the Florida Research and Development Center near West Palm Beach, Fla.

HAZARDS OF LIQUID HYDROGEN IN RESEARCH AND DEVELOPMENT FACILITIES - Atlantic Research Corporation, Alexandria, Va., ASD-TDR-62-1027 (Dec. 1962), Contr. No. AF 33(657)-8952, 75 pp 1 fig 4 tab 27 ref.

It is the objective of this report to establish the hazards associated with the use of liquid hydrogen in research and development facilities, and to review the applicable techniques of hazard prevention and control. For this purpose the physical and chemical properties of hydrogen have been summarized with emphasis on the relation of these properties to combustion and detonation processes, and information has been collected on experiences and practices in numerous facilities. Hazards comprise the possibility of pressure rupture of containers, initiation of flammable mixtures formed by release of hydrogen due to vessel failure or other causes, and explosion of hydrogen and contaminating oxygen under cryogenic conditions. It is shown that the theory of chemical reaction provides a complete understanding of the combustion and detonation characteristics of hydrogen-oxygen systems and defines the chemical and physical requirements for inhibition and control of combustion and detonations. Experiences and practices in research and development facilities have been analyzed, general safety procedures have been suggested and subjects warranting further investigation have been determined.

HYDROGEN - Atomic Energy Commission, Washington, D. C., Safety & Fire Protection Bull. No. 5 (Aug. 1956) 10 pp 3 fig 16 ref.

The Atomic Energy Commission and its contractors have experienced a number of serious accidents involving hydro-

gen gas. These have included, variously, hydrogen cooled equipment, by-product hydrogen, hydrogen generated as a result of water-metal reactions, process hydrogen, and the pressure-testing of hydrogen containers. This bulletin covers properties, uses, handling and explosion characteristics of gaseous and liquid hydrogen, and provides various listings of precautions and safe practices to be observed in working with hydrogen.

HAZARDS DUE TO HYDROGEN ABOARD A SPACE VEHICLE - Caras, Gus. J. - Redstone Scientific Information Center Rept. 291 (Sept. 1964) 17 pp 23 ref.

This bibliography consists of 23 annotated references on the subject of hazards to space vehicles as a result of hydrogen leaks. The references treat such topics as hydrogen-leak detection and suppression of fires and explosions. Since the subject of hydrogen safety is rather broad, an attempt was made to include only those references which were related in some way to safety of space cabins or similar compartments.

HYDROGEN - Compressed Gas Association, Inc., New York (1955) Pamphlet G-5 15 pp 3 fig.

This pamphlet is one of a series of publications compiled by the Compressed Gas Association, Inc. to satisfy the demand for information relative to the transportation, handling and storage of compressed gases.

In this pamphlet an attempt has been made to present general information regarding the characteristics of hydrogen and its handling.

TENTATIVE STANDARD FOR LIQUEFIED HYDROGEN SYSTEMS AT CONSUMER SITES - Compressed Gas Association, Inc., New York - Pamphlet G-5.2T (Aug. 1965), 8 pp 2 tab.

This standard covers the general principles recommended for the installation of liquefied hydrogen systems on consumer premises.

A STUDY OF THE HAZARDS IN THE STORAGE AND HANDLING OF LIQUID HYDROGEN - Cassutt, L. H., Maddocks, F. E. and Sawyer, W. A. - Advances in Cryogenic Engineering 5, 55-61 (Proc. of 1959 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) 3 fig 11 ref.

This paper describes the results of a research program initiated to develop realistic safety criteria for the storage and handling of liquid hydrogen. Such criteria could bring about substantial savings in the capital equipment costs of production and storage facilities and could point out safety devices which would prevent major losses. Also a reduction in the required area for a production or storage facility might be effected, thus decreasing the costs of such facilities.

ELECTROSTATIC HAZARDS ASSOCIATED WITH THE TRANSFER AND STORAGE OF LIQUID HYDROGEN - Cassutt, L., Biron, D. and Vonnegut, B. - Advances in Cryogenic Engineering 7, 327-35 (Proc. of 1961 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1962) Paper H-1, 7 fig 1 tab 17 ref.

This paper summarizes the results of an experimental program to investigate the potential hazards of static charge generation and accumulation in well-grounded liquid hydrogen storage and transfer systems. Results of previous investigations of hydrogen hazards indicate that, because of the very low ignition energy requirements and wide range of flammability for mixtures of hydrogen and air (or oxygen), extreme care must be taken to remove all possible ignition sources from hydrogen facilities. The elimination of static electricity as an ignition source, however, required greater knowledge than was available concerning the conditions governing its occurrence in liquid hydrogen service.

PROPERTIES OF LIQUID HYDROGEN - Corruccini, R. J. - Paper presented at meeting of International Institute of Refrigeration - Commission I, Grenoble, France (June 8, 1965) 53 pp 14 fig 6 tab 64 ref.

In this paper a survey is made of the available knowledge of those physical properties of liquid hydrogen that are of technological importance. First, some of the physics that explain the special characteristics of hydrogen are presented. Then, a catalog of the major sources of data and extracts of these data are given. Finally, some special topics are discussed that have not been adequately dealt with in previous surveys.

LIQUID HYDROGEN ENGINEERING INSTRUMENTATION - Flynn, T. M. - Paper presented at meeting of International Institute of Refrigeration - Commission I, Grenoble, France (June 8, 1965) 38 pp 15 fig 12 ref.

Prudent liquid hydrogen engineering requires the measurement of both extensive and intensive properties of the cryogenic liquid. Transducers are required for liquid level (quantity), both point and continuous systems, and mass rate systems. In addition, there must be transducers of pressure, temperature, density, and occasionally, quality. This paper discusses some of the devices and practices currently used for the measurement of pressure, temperature, flow rate, and liquid level in liquid hydrogen systems.

LIQUID HYDROGEN TECHNOLOGY - General Dynamics/Astronautics, San Diego, Calif. - Rept. No. AE62-0774 (Sept 1962) 294 pp 143 fig 24 tab 144 ref. STIF N64-10128.

This report summarizes a continuing study of liquid hydrogen technology being conducted by General Dynamics/Astronautics in company-funded research and in development of the hydrogen-fueled Centaur space vehicle for the National Aeronautics and Space Administration. The report is intended to serve as a standard reference on liquid hydrogen properties, handling and storage with primary emphasis on space vehicle applications. The following 14 areas are included: manufacture, transportation, hydrogen safety, materials compatibility, cryogenic insulation, transfer, cryogenic measurements, propulsion methods, sloshing, vortexing, propellant heating, zero-gravity behavior, space storage, properties.

SAFETY PROBLEMS AND SAFETY CODES CONCERNING LIQUID HYDROGEN AND LIQUID HELIUM - Edesky, F. J. Los Alamos Scientific Laboratory, Los Alamos, New Mexico. Presented at the XIIth International Congress of Refrigeration, Madrid, Spain (Sept. 1967) 33 pp 2 fig 5 tab 33 ref.

Safety problems which occur in the storage and handling of liquid hydrogen and liquid helium include material selection, contamination, pressure relief and stress analysis. Proper material selection requires optimization of a number of factors, only one of which is the metal ductility at the use temperature. Consideration of temperature gradients during the cooldown process has shown that stress contributions from this source are important and must be included in the overall system stress analysis. Careful control must be exercised over contaminants since they can accumulate over a period of time which could result in line plugging or the formation of explosive or detonable mixtures.

Safety codes are being developed for liquid hydrogen. Safety restrictions which exist cover shipping procedures, production purities, minimum distances between storage vessels and between storage vessels and other facilities. Distance standards are not necessarily uniform as this is an evolving technology. Significant experience to date with large quantities of liquid hydrogen permits a high level of confidence in the use of this material.

PRINCIPLES FOR SAFE HANDLING OF LIQUID HYDROGEN - Grumer, J., Strasser, A., and Van Meter, R. A. U. S. Bur. Mines, Dept. of Interior, Explosives Research Lab., Pittsburgh, Pa., Tech. Rept. (June 1967) 34 pp 2 tab 31 ref.

Recommendations for the safe handling of liquid hydrogen, based on an extensive survey of the literature and of safety practices at various installations, are presented. Consideration is given to fires and explosions which constitute the most serious hazard. Measures for fire control and safe practice in hydrogen storage and transfer are discussed. Importance of prompt and accurate detection of hydrogen leaks is stressed, together with the need for better detection devices. Finally, emphasis is placed on the fact that there is no substitute for well-trained, adequately supervised and safety-conscious personnel.

SAFE HANDLING OF LIQUID HYDROGEN - Grumer, J., Strasser, A., and Van Meter, R. A. - Cryogenic Eng. News 2, 60-63 (Aug. 1967).

The greatly increased use of hydrogen in recent years has led to a corresponding increase in the need for sound safety practices and precautions. As part of a broad program sponsored by the Space Nuclear Propulsion Office, the Bureau of Mines investigated hazards associated with liquid hydrogen operations - listing eighty-three recommendations in this article to eliminate such hazards.

DESIGNING FOR SAFETY IN HYDROGEN BUBBLE CHAMBERS - Hernandez, H. P., Mark, J. W. and Watt, R. D. - Rev. Sci. Instr. 28, 528-35 (July 1957).

The principal hazards of operating a liquid hydrogen bubble chamber are failure of equipment (due to overpressure) and uncontrolled escape of hydrogen gas, which may cause an explosion. If safety conditions are incorporated in planning from the job beginning, components can be designed to reduce or eliminate the probability of accidents arising from the known hazards. The degree of safety, the hydrogen safety-vent system, and the hazards and operation of the liquid hydrogen bubble chambers at UCRL are discussed.

SAFETY REVIEW: LIQUID HYDROGEN SERVICING SYSTEM PAD 37B SATURN C-1 - CAPE CANAVERAL, FLORIDA - Himmelberger, F. and Vander Arend, P. C. - Air Products & Chem., Inc., Allentown, Pa., Contr. No. NAS 8-1546, 35 pp 5 fig 1 tab 9 ref.

This written Safety Review discusses the underlying principles of liquid hydrogen safety in Part I, and then, in Part II, defines how these principles have been applied to the design of the Liquid Hydrogen Servicing System on Pad 37B (Cape Canaveral, Florida). Since the safety aspects of fuel handling are well known, greater emphasis has been placed on the cryogenic factors governing liquid hydrogen safety.

HANDLING LIQUID HYDROGEN ON A PILOT-PLANT SCALE - Laquer, H. L. - Advances in Cryogenic Engineering 5, 85-94 (Proc. of 1959 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) 12 fig 4 ref.

The following discussion summarizes experience obtained during the last three years while handling liquid hydrogen in 5500 - liter batches. The work was not an objective in itself, nor is it complete or exhaustive, but was done only coincidentally while developing and operating a liquid hydrogen-cooled electromagnet. The quantities of liquid handled are small by missile standards, but the problems encountered on this pilot-plant scale are much nearer to those of large-scale usage than those of normal laboratory usage. The scaling of volumes would be from 1 gal. in the laboratory to 1000 gal. in our magnet to perhaps 100,000 gal. for missile testing.

PROCEDURES FOR THE DESIGN AND OPERATION OF HAZARDOUS RESEARCH EQUIPMENT - U. S. A. E. C., Lawrence Radiation Lab., Univ. of Cal., Berkeley, Cal., UCRL-9711 (Oct. 1961) 73 pp 12 fig 7 ref.

This manual sets forth the procedures for the safe design and operation of hazardous research equipment, such as hydrogen targets, bubble chambers, high-pressure gas apparatuses, and the like. These procedures are in addition to the LRL general safety requirements set forth by the Plant Safety and Emergency Service Department.

PRECAUTIONS AND SAFE PRACTICES FOR HANDLING LIQUID HYDROGEN - Linde Company, Div. of Union Carbide Corp., Publication F-9914.

The purpose of this booklet is to outline the basic techniques for the safe handling of liquid hydrogen.

INTERIM REPORT ON AN INVESTIGATION OF HAZARDS ASSOCIATED WITH LIQUID HYDROGEN STORAGE AND USE - Little, Arthur D., Inc. (Jan. 1959) Contr. AF 18 (600)1687, 92 pp 24 fig 5 tab 14 ref.

Over the years there has been accumulated in various quarters a considerable, but nevertheless limited, experience with the hazards associated with handling of liquid hydrogen in gallon quantities. There have been several explosions and accidents reported (appendix A) - some explained and others from unknown causes - but there has never been a satisfactory basis for predicting the magnitude of hazard associated with the large-scale production, storage and handling of liquid hydrogen.

It was the purpose of the current assignment to establish through an experimental program the criteria for storing and transporting large quantities of liquid hydrogen and - on the basis of the findings - to recommend realistic quantity-distance relationships and safe-handling procedures.

FINAL REPORT ON AN INVESTIGATION OF HAZARDS ASSOCIATED WITH THE STORAGE AND HANDLING OF LIQUID HYDROGEN - Little, Arthur D., Inc. (March 1960) Contr AF 18(600)-1687.

The material presented in this report summarizes the efforts of a program designed to provide a more satisfactory basis for establishing reasonable safety procedures for the storage and transportation of large quantities (up to 100,000 lb.) of liquid hydrogen. Experience of others, including accident records and safety practices, were studied and an experimental program to gather much needed additional information was conducted. Details of the experimental program are presented in Appendix A. The conclusions and recommendations concerned with safe handling and proper storage criteria derived from the results of the program are presented in the main body of the report.

FINAL REPORT ON AN INVESTIGATION OF HAZARDS ASSOCIATED WITH THE STORAGE OF LIQUID HYDROGEN IN CLOSE PROXIMITY TO LIQUID OXYGEN AND RP-1 (CONFIDENTIAL) - Little, Arthur D., Inc., (July 1960) Contr. AF 18(600)1687.

LIQUID HYDROGEN SAFETY MANUAL - Little, Arthur D., Inc., Detachment No. 2, (Oct. 1959) Cont. AF 18 (600)1687.

This document covers general properties, hazards, safety measures, transfer and storage, shipping of, and recommended safety instruction format for use with, liquid hydrogen.

HYDROGEN HANDBOOK. A COMPILATION OF PROPERTIES, HANDLING AND TESTING PROCEDURES, COMPATIBILITY WITH MATERIALS, AND BEHAVIOR AT LOW TEMPERATURES - Little, Arthur D., Inc., Cambridge, Mass., AFFTC TR 60-19 (April 1960) (Subcontract to Parker Aircraft Co., Los Angeles, Calif.) Contr. No. 33(616)6710; 246 pp 89 fig 20 tab 29 ref. DDC AD 242 285.

This report summarizes (1) experience with and the available technical information on the development of two proto-

type valves, one for a cryogenic gas service and the other for a cryogenic liquid service (these valves are under development by Parker Aircraft Co. for a government agency); (2) bibliographical information on the physical and mechanical properties of specific construction materials for a temperature range of -420°F to +200°F (these materials include some austenitic stainless steels and Teflon plastics); (3) the thermodynamic properties of helium, hydrogen, and nitrogen fluids with which the valves may be used or tested; (4) the hazards associated with the transportation and storage of hydrogen and with its use for testing the prototype valves for leakage across the seals; and (5) the sources and availability of hydrogen, and the Los Angeles regulations that apply to its transportation and use.

HANDBOOK FOR HYDROGEN HANDLING EQUIPMENT - Little, Arthur D., Inc., WADC TR 59-751 (May 1961) Contr. AF 33(616)5641, 583 pp 226 ref. DDC AD 235 123.

This handbook provides engineering data concerning the most adequate, safe, and economical procedures and equipment for liquid hydrogen storage, transfer, and ground servicing systems. The engineering data developed centers on the requirements for (1) storage vessels, (2) transfer lines, (3) pumping systems, (4) valves, (5) instruments, and (6) recondensing systems. Results, conclusions and recommendations are reported in separate chapters classified in accordance with the above listed hardware items.

STORAGE, TRANSFER, AND SERVICING EQUIPMENT FOR LIQUID HYDROGEN - Little, Arthur D., Inc., WADC Tech Rept. 59-386 (July 1959) Contr. AF 33(616)5641, 772 pp 237 ref.

The purpose of this study is to provide engineering data concerning the most adequate, safe, and economical procedures and equipment for liquid hydrogen storage, transfer, and ground servicing systems.

Investigations have centered on the requirements for (1) storage vessels, (2) transfer lines, (3) pumping systems, (4) valves, (5) instruments, and (6) recondensing systems. Results, conclusions and recommendations are reported in separate chapters classified in accordance with the above listed hardware items.

STORAGE, SERVICING, TRANSFER AND HANDLING OF LIQUID HYDROGEN - Little, Arthur D., Inc., AFFTC TR 61-18 (May 1961) Contr. No. AF 33(616)7330, 162 pp 26 ref.

This report documents the results of investigations into six technical areas pertaining to the handling of large quantities of liquid hydrogen. The engineering data developed are reported in six separate sections. Section I establishes the current availability and specifications for liquid hydrogen pumps and cites the operational experience reported by the major users of these items. Section II sets forth a tested method for predicting the hydrogen gas required for the pressurized transfer of liquid hydrogen. Section III recounts the known facts relating to the safety and reliability of hydrogen gas cylinders as used in typical operations for the pressurized transfer of liquid hydrogen. Section IV presents the design

specifications and performance characteristics of gravity-fed and boosted pressure-fed vaporizers for liquid hydrogen transfers established as a result of an integrated program of theoretical analysis and tests. Section V includes an economic comparison of systems using pumps, hydrogen vaporizers and high pressure hydrogen gas bottles to transfer liquid hydrogen. Section VI presents the results of a further investigation of the single parting line coupling for vacuum-jacketed transfer lines originally reported in WADC TR 59-386.

HYDROGEN SAFETY MANUAL - Advisory Panel on Experimental Fluids and Gases NASA - Lewis Research Center, Cleveland, Ohio NASA TMX-52454 (1968) 79 pp 7 fig 4 tab.

This manual is designed to cover most aspects of hydrogen handling and usage. Both personnel and equipment are concerned. It is the intent to present here acceptable hydrogen standards and practices for minimum safety requirements only. More extensive safety precautions should be employed when there is extra hazard, as in highly-congested areas or in operations with equipment that has little safety margin.

STANDARD FOR GASEOUS HYDROGEN SYSTEMS AT CONSUMER SITES - National Fire Protection Association, Rept. No. 567 (1963) 11 pp.

This Standard covers the general principles recommended for the installation of gaseous hydrogen systems on consumer premises. It covers requirements for gaseous hydrogen systems including design, location, operation and maintenance. The Standard does not apply to hydrogen manufacturing plants or other establishments operated by the hydrogen supplier or his agent for the purpose of storing hydrogen and refilling portable containers, trailers, mobile supply trucks or tank cars.

LIQUID HYDROGEN HANDLING AND SAFETY - Maddocks, F. E. - Little, Arthur D., Inc., Santa Monica, Calif. (No date) 29 pp 16 fig 1 tab 12 ref.

The history of liquid hydrogen production is reviewed from the first liquefaction in liter quantities until current production of many tons per day. Some of the interesting physical and thermal properties of the liquid are discussed with particular emphasis on those of most interest to the missile and space designer. A typical process for liquid hydrogen production is described and the growth of the U. S. production capacity is reviewed. A description of current storage vessel and transfer line design practice is given. Some of the limitations of metals, both ferrous and non-ferrous, and non-metals when used at liquid hydrogen temperature are pointed out. The paper concludes with a detailed review of safety problems including detonation, deflagration and radiation hazards as well as recommendations for safe handling of the material.

SAFETY INSTRUCTION AND SAFETY GUIDE FOR HANDLING GASEOUS AND LIQUID HYDROGEN AT THE BOULDER LABORATORIES - Natl. Bur. Standards Memo. Rept. No. CM-4 (Jan. 1960) 30 pp 12 ref.

The Safety Instruction outlines organizational responsibilities and application of the Safety Guide for the safe use of hydrogen in the Boulder Laboratories. The Safety Guide outlines safety considerations pertinent to the various situations and conditions in which liquid and gaseous hydrogen is used in the Boulder Laboratories. The subject matter covered is developed with the intent of increasing personnel awareness of the hazards involved in handling hydrogen and pointing out practical methods of minimizing the hazards. It is not intended to prohibit or inhibit the use of hydrogen for experimental purposes.

HANDLING OF LIQUID AND GASEOUS HYDROGEN - Schmidt, A. F. - Presented in the panel on Handling of Propellants and Gases, SAE National Aeronautic Meeting, Los Angeles, Calif., (Oct. 11, 1960) 7 pp.

Problems associated with the use of hydrogen are discussed in the light of present-day cryogenic fuel handling technology; an examination of several characteristics of hydrogen is made - specifically those concerned with its ignition and subsequent detonation or deflagration.

LIQUID HYDROGEN - Schmidt, E. W. - Raketentechnik und Raumfahrtforschung 5, 24-26 (1961); also available in English translation as: Tech. Memo. 1028, Feltman Research Laboratories, Picatinny Arsenal, Dover, New Jersey (August 1962) 18 pp 1 fig 1 tab 9 ref.

The problems in the manufacture, storage, and handling of liquid hydrogen are examined. Cited are the operations of a number of American firms, including Pratt and Whitney. The primary problem discussed is the loss through evaporation and the pipes, valves, fittings, and storage tanks designed to counteract this loss in order that the liquid hydrogen may be used to better advantage as a rocket propellant.

CRYOGENIC HYDROGEN - Pratt and Whitney Aircraft Div., United Aircraft Corporation - Booklet S-945 (Mar. 1965).

Engineers at Pratt and Whitney Aircraft's Florida Research and Development Center have handled millions of gallons of liquid hydrogen during years of active experience with this material. Some of the techniques they have learned are published in the hope they may be of interest to those associated with projects employing this fuel. Sections of the booklet deal with preparation, storage, handling, physical and thermodynamic properties of hydrogen.

THE SAFE HANDLING OF LIQUID HYDROGEN - Scharle, W. J. - Trans. Inst. Chem. Engrs. (London) 185, CE 16-24 (Jan.-Feb. 1965).

This paper presents an up-to-date summary of current practices in the U. S. A. on the safe storage and handling of liquid hydrogen. Present yearly consumption in the U. S. A. is over 50 million gallons.

A brief review of the properties of hydrogen and general procedures for safe handling is given. Quantity-distance relationships for liquid-hydrogen storage tanks, both for pro-

duction facilities and missile test stands are given. Safe methods for disposal of varying quantities of liquid hydrogen require special consideration, and criteria are presented.

Quality-control aspects for liquid hydrogen are becoming more rigid. The hazards of oxidants present in liquid hydrogen are discussed. A brief description of purity requirements is given along with a standard method for sampling and analysis.

A description of methods of transfer and transfer equipment is given.

THE STORAGE AND HANDLING OF HYDROGEN WITH SAFETY - Stoll, A. P. - Trans. Inst. Chem. Engrs. (London) 185, CE 11-16 (Jan. - Feb. 1965).

Safety precautions during the handling of hydrogen, whether liquid or gaseous, are dictated by the relative ease of ignition of hydrogen whether in air or in oxygen. Considerable pressures can be built up if reaction of combustion is contained and protection is primarily based on preventing the formation of explosive mixtures of hydrogen with air both inside and outside the equipment. Further, probable sources of ignition should be segregated or eliminated altogether. The effects of burning liquid hydrogen and methods of dealing with such fires are discussed.

The storage and handling of liquid hydrogen at - 253°C requires special techniques, which are by now well established. Details are given of how liquid hydrogen should be handled in the laboratory, showing that with due care such work can be carried out quite safely.

TECHNOLOGY AND USES OF LIQUID HYDROGEN - Scott, R. B., Denton, W. H., Nicholls, C. M. (Editors) Pergamon Press, Oxford, England (1964) 415 pp.

This book is a compilation of advanced technical information from the foremost laboratories of the United States, England, Switzerland, and West Germany on the production and uses of liquid hydrogen. It covers technical aspects of production of hydrogen gas for liquefaction, hydrogen liquefiers, properties of hydrogen, insulation requirements for storage and transport, safety practices, and uses ranging from refrigerant to rocket fuel.

SAFE ELECTRICAL INSTALLATIONS IN ATMOSPHERES CONTAINING HYDROGEN - Scott, R. W. - Chem. Eng. (Mar. 2, 1964) 4 pp.

Areas where extremely flammable and explosive hydrogen is used are designated by the National Electrical Code as Class I, Group B, Div. 1 hazardous locations. Electrical equipment used in these areas should be designed in accordance with Underwriter's Laboratories (U/L) requirements. If not, there is a good possibility that an electrical spark or arc caused by this equipment might touch off a massive explosion and fire.

This article reviews the hazards of hydrogen, intrinsically safe equipment, purged or pressurized systems, and explosion-proof enclosures.

HYDROGEN DETECTORS - Strasser, A., Liebman, I. and Harris, S. R. - Cryogenic Eng. News 2, 16-18, 20

(Dec. 1967).

The greatly increased use of hydrogen in recent years has led to a corresponding increase in the need for reliable hydrogen detectors. As part of a broad program about the safe use of hydrogen, sponsored by the Space Nuclear Propulsion Office, the Bureau of Mines investigated the performance of a number of hydrogen detectors. The results of this investigation are presented here. Its purpose was to determine the limitations and potentialities of the kinds of detectors examined; it was not intended to provide official approval or disapproval of the instruments.

LARGE-SCALE PRODUCTION, HANDLING, AND STORAGE OF LIQUID HYDROGEN - Vander Arend, P. C. - Advances in Cryogenic Engineering 5, 49-54 (Proc. of 1959 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) Paper A-4, 1 fig.

With the engineering, design and construction of large-scale liquid hydrogen production facilities under direction of the Air Force, liquid hydrogen has become a propellant available on a sustained and economic basis. A short review of the past few years shows how tremendous the progress in hydrogen technology has been.

CONTROL OF LIQUID HYDROGEN HAZARDS AT EXPERIMENTAL FACILITIES: A REVIEW - Weintraub, A. A. - AEC Health and Safety Laboratory Rept. 160 (May 1965) 249 pp 26 fig 4 tab 60 ref.

The hazards of liquid hydrogen at experimental facilities are described and current techniques are presented for their control. As background, the uses of hydrogen, its general properties (Appendix A notes specific properties) and the design and materials for liquid hydrogen systems are discussed. Flame propagation, deflagration and detonation, ignition in free space and under confined conditions are considered under fire and explosion hazards.

The techniques of hazard control are examined in relation to hydrogen detection systems, ventilation, liquid hydrogen dump systems, flaring, ignition sources, explosion suppression, inerting, fire control and personnel exposures. Storage, transfer and transportation are also discussed. Appendix B contains case histories of hydrogen accidents. Two of the liquid hydrogen safety guides, cited among the 60 references, are reproduced in Appendix C.

THE APPLICATION OF COMMERCIAL ELECTRICAL EQUIPMENT TO LOCATIONS WHERE HYDROGEN GAS MAY EXIST IN QUANTITIES SUFFICIENT TO PRODUCE EXPLOSIVE OR IGNITABLE MIXTURES - Woodard, K. A. - Advances in Cryogenic Engineering 1, 144-47 (Proc. of 1954 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) Paper C-6.

The purpose of this paper is to summarize the type of electrical equipment and methods of installation chosen for industrial plants and miscellaneous equipment installations used in processing liquid or gaseous hydrogen. An attempt has been made to set up systematic design procedures built around the National Electrical Code wherever

practicable. This paper is not intended to be used as a rule book, but only as a guide and as a record of past problems and their solutions in order to expedite future design problems and to help insure the safest performance possible.

An expanded version of this paper was written in 1955 by the author for the Stearns-Roger Manufacturing Company, Denver, Colo., under the title: A SAFETY GUIDE FOR THE APPLICATION OF COMMERCIAL ELECTRICAL EQUIPMENT IN LIQUID HYDROGEN FACILITIES.

HAZARDS IN THE HANDLING OF CRYOGENIC FLUIDS - Zabetakis, M. G. - Advances in Cryogenic Engineering 8, 236-41 (Proc. of 1962 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1963) Paper E-1, 1 tab 28 ref.

The general safety precautions to be used in the liquefaction, handling, storage, and use of compressed gases are reviewed briefly. Physiological effects of cryogenic fluids and their behavior in air and in contact with various structural materials are considered in some detail. The usual hazards associated with inert, combustibles and oxidants are treated in terms of fluids that are initially at low temperatures.

RESEARCH ON THE HAZARDS ASSOCIATED WITH THE PRODUCTION AND HANDLING OF LIQUID HYDROGEN - Zabetakis, M. G. and Burgess, D. S. - U.S. Bur. Mines, WADD Tech. Rept. 60-141 (June 1960) Contr. AF 33(616) 58-5, 76 pp 39 fig 10 tab 22 ref.

The use of liquid hydrogen as a high-energy fuel introduces numerous hazards not ordinarily associated with the use of other more conventional fuels. These hazards are attributable to the unique properties of hydrogen in the liquid and gas states. Since little work has been conducted on explosion and related hazards of cryogenic fuels, a research program was undertaken to obtain basic data on such dangers for hydrogen and other combustibles under these conditions. The data were used to outline emergency procedures for protecting personnel and equipment when an accident spillage of liquid hydrogen occurs and to establish a quantity-distance table for the storage of this fuel.

EXPLOSION HAZARDS OF LIQUID HYDROGEN - Zabetakis, M. G., Furno, A. L., and Martindill, G. H. - Advances in Cryogenic Engineering 6, 185-94 (Proc. of

1960 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1961) Paper D-2, 7 fig 1 tab 12 ref.

The use of liquid hydrogen as a low temperature fluid and as a high energy fuel presents explosion hazards not ordinarily encountered with other cryogenic fluids. These hazards arise because of the many unique properties of this combustible in both the liquid and gaseous states. A program was started in 1958 by the Federal Bureau of Mines to determine the nature and extent of the above hazards. The data obtained from this program have been used to formulate emergency procedures and safeguards for the prevention of a disaster following the accidental spillage of liquid hydrogen. These data, emergency procedures and safeguards are summarized in this report.

RESEARCH ON THE HAZARDS ASSOCIATED WITH THE USE OF LIQUID HYDROGEN IN BUBBLE CHAMBERS - Zabetakis, M. G., Furno, A. L. and Perlee, H. E. - U.S. Bur. Mines, Dept. of Interior, Explosives Research Lab., Pittsburgh, Pa., Final Rept. No. 3861 (May 1962) 53 pp 24 ref.

In the present study, certain of the hazards associated with the use of liquid hydrogen in enclosed spaces were investigated in some detail. Of particular interest were those hazards associated with the use of the liquid in bubble chambers. These could arise from the failure of a glass window, container, vacuum tank, vent system, etc. Accordingly, special efforts were made to determine the nature of glass window failures, the results to be expected from the impingement of glass fragments on materials found in bubble chamber vacuum tanks, the performance of vent systems, the desirable characteristics of hydrogen gas detection systems, the method of ignition of hydrogen-air mixtures, and finally, the results to be expected from the ignition of such mixtures in enclosed spaces.

HAZARDS IN USING LIQUID HYDROGEN IN BUBBLE CHAMBERS - Zabetakis, M. G., Furno, A. L. and Perlee, H. E. - U.S. Bur. Mines, Dept. of Interior, Explosives Research Lab. Pittsburgh, Pa., RI 6309 (1963) 39 pp 27 fig 29 ref.

(Similar in content to preceding report; slightly different arrangement of material.)

PHYSICAL PROPERTIES OF HYDROGEN

PROPERTY VALUES OF NORMAL
HYDROGEN AT SELECTED CONDITIONS

Nomenclature and Notations

TP = Triple Point

NBT = Normal Boiling Temperature (and 1 atm.)

NTP = Normal Temperature and Pressure
(70°F, 14.7 psia)

273.15°K* = 0°C = 32°F = 491.67°R

The term "mole" as used here means "gm-mole."

a. The Advisory Committee on Thermometry of the International Committee on Weights and Measures has agreed on 20.384°K as the present most probable value of thermodynamic temperature for the normal boiling temperature of normal hydrogen; see Brickwedde, F. G., "International Practical Temperature Scale," Physics Today 16, 24-26 (1963).

*Changes have been made to correct data to this scale where necessary.

b. C_p (vapor)_{NBT} could be used here.

c. Extrapolated value.

d. C_s (liquid)_{NBT} could be used here.e. Assuming b above is done, the value of C_p/C_v (liquid)_{NBT} is then 1.6.

f. Value taken at 20°K (-423.67°F).

Property Values of Normal Hydrogen at Selected Conditions				
Property		Value	Reference	
Molecular Weight		2.01594	1	
Triple Point Values	Temperature, °K	13.95	2, 3, 4	
	Pressure, mm Hg	54.0	3, 4	
	Density, mole/cc	0.0430	3, 4	
	Liquid	0.0383	3, 4	
	Vapor	0.0000631	4	
Normal Boiling Values	Temperature (T _b), °K	20.38	2, 3, 4	
	Density, mole/cc	0.0352	2, 3, 4	
	Vapor	0.000661	4	
Critical Values	Temperature, °K	33.18	2, 3, 4	
	Pressure, mm Hg	5865	3, 4	
	Density, mole/cc	0.0149	2, 3, 4	
One Liter Liquid (NBT) Equivalents	Weight, kg	0.0710	1, 2, 3	
	Volume of NBT	53.3	2, 3, 4	
	Gas, liters	850	2, 3, 5	
Equivalent Volumes of Gas per Volume of Liquid (NBT)	NBT	53.3	2, 3	
	NTP	850	2, 3, 5	
Heat of Fusion, Cal/mole	TP	28.0	3, 4	
Heat of Vaporization, Cal/mole	NBT	216	3, 4	
Specific Heat Cal/mole-°K	C_s	Solid TP	1.37	3
		Liquid NBT	4.6	3
		Vapor NBT		b
	C_v	Liquid NBT	2.9	3
		Vapor NBT	3.0	c
		Gas NTP	4.90	6
	C_p	Liquid NBT		d
		Vapor NBT	5.8	3, 4
		Gas NTP	6.89	6, 4
Specific Heat Ratio	C_p/C_v	Liquid NBT		e
		Vapor NBT	1.9	3
		Gas NTP	1.41	6
Thermal Conductivity K Cal/cm-sec-°K		Liquid NBT	0.000284	7
		Vapor NBT	0.000038	6, 4
Viscosity μ Gm/cm-sec		Gas NTP	0.000427	6, 4
		Liquid NBT	0.000135	8, 3
		Vapor NBT	0.0000109	3, 6, 4
		Gas NTP	0.0000884	3, 6, 4

Property Values of Normal Hydrogen at Selected Conditions				
Property		Value	Reference	
Molecular Weight		2.01594	1	
Triple Point Values	Temperature, °F	-434.56	2, 3, 4	
	Pressure, psia	1.04	3, 4	
	Density, lb/ft ³	5.41	3, 4	
	Liquid	4.82	3, 4	
	Vapor	0.00794	4	
Normal Boiling Values	Temperature (T _b), °F	-422.99	2, 3, 4	
	Density, lb/ft ³	4.43	2, 3, 4	
	Vapor	0.0832	4	
Critical Values	Temperature, °F	-399.95	2, 3, 4	
	Pressure, psia	190.8	3, 4	
	Density, lb/ft ³	1.88	2, 3, 4	
One Gallon Liquid (NBT) Equivalents	Weight, lb	0.592	1, 2, 3	
	Volume of NBT	7.12	2, 3, 4	
	gas, ft ³	113.7	2, 3, 5	
Equivalent Volumes of Gas per Volume of Liquid (NBT)	NBT	53.3	2, 3	
	NTP	850	2, 3, 5	
Heat of Fusion, Btu/lb	TP	25.0	3, 4	
Heat of Vaporization, Btu/lb	NBT	193	3, 4	
Specific Heat Btu/lb-°F	C_s	Solid TP	0.679	3
		Liquid NBT	2.3	3
		Vapor NBT		b
	C_v	Liquid NBT	1.4	3
		Vapor NBT	1.5	c
		Gas NTP	2.43	6
	C_p	Liquid NBT		d
		Vapor NBT	2.9	3, 4
		Gas NTP	3.41	6, 4
Specific Heat Ratio	C_p/C_v	Liquid NBT		e
		Vapor NBT	1.9	3
		Gas NTP	1.41	6
Thermal Conductivity K Btu/hr-ft-°F		Liquid NBT	0.0687	7
		Vapor NBT	0.0092	f
Viscosity μ Centipoise		Gas NTP	0.103	6, 4
		Liquid NBT	0.0135	8, 3
		Vapor NBT	0.00109	f
		Gas NTP	0.00884	3, 6, 4

PROPERTY VALUES OF
PARAHYDROGEN AT
SELECTED CONDITIONS

Nomenclature and Notations

TP = Triple Point

NBT = Normal Boiling Temperature (and 1 atm.)

NTP = Normal Temperature and Pressure
(70°F, 14.7 psia)

273.15°K* = 0°C = 32°F = 491.67°R

The term "mole" as used here means "gm-mole."

a. Calculated with the assumption that the volume change on fusion is the same for normal and parahydrogen with the value of ΔV from reference 3.

b. The Advisory Committee on Thermometry of the International Committee on Weights and Measures has agreed on 20.267°K as the present most probable value of thermodynamic temperature for the normal boiling temperature of parahydrogen; see Brickwedde, F. G., "International Practical Temperature Scale," Physics Today 16, 24-26 (1963).

*Changes have been made to correct data to this scale where necessary.

c. C_p (vapor)_{NBT} could be used here.

d. C_v (gas)_{NTP} of normal hydrogen could be used here.

e. C_p (gas)_{NTP} of normal hydrogen could be used here.

f. Assuming c and d above are done, the value for C_p/C_v (gas)_{NTP} is then 1.41.

g. Value taken at 20°K (-423.67°F).

h. The difference between the viscosities of normal and parahydrogen is less than 1% from 15° to 90°K; see references 16 and 17. Therefore μ (gas)_{NTP} of normal hydrogen could be used here assuming the trend of normal-to-para difference.

Property Values of Parahydrogen at Selected Conditions					
Property			Value	Reference	
Molecular Weight			2.01594	1	
Triple Point Values	Temperature, °K		13.803	2, 9	
	Pressure, mm Hg		52.8	9	
	Density, mole/cc	Solid	0.0429	a	
		Liquid	0.0382		2, 9, 14
		Vapor	0.0000624		9, 14
Normal Boiling Values	Temperature (T _b), °K		20.268		2, 9
	Density, mole/cc	Liquid	0.03511	2, 9, 14	
		Vapor	0.000664	9, 14	
Critical Values	Temperature, °K		32.976	9, 14	
	Pressure, mm Hg		9697	9, 14	
	Density, mole/cc		0.0156	9, 14	
One Liter Liquid (NBT) Equivalents	Weight, kg		0.07078	9	
	Volume of Gas, liters	NBT	52.9	2, 9	
		NTP	848	2, 6, 9	
Equivalent Volumes of Gas per Volume of Liquid (NBT)	NBT		52.9	2, 9	
	NTP		848	2, 6, 9	
Heat of Fusion, Cal/mole		TP	28.1	10	
Heat of Vaporization, Cal/mole		NBT	214.8	9, 10, 14	
Specific Heat Cal/mole-°K	C _s	Solid	TP	1.36	3, 10
		Liquid	NBT	4.63	12
		Vapor	NBT		c
	C _v	Liquid	NBT	2.72	13, 14
		Vapor	NBT	3.13	14
		Gas	NTP		d
	C _p	Liquid	NBT	4.7	14
		Vapor	NBT	5.86	14
		Gas	NTP		e
Specific Heat Ratio	C _p /C _v	Liquid	NBT	1.72	13, 14
		Vapor	NBT	1.902	14
		Gas	NTP		f
Thermal Conductivity K Cal/cm-sec-°K	Liquid	NBT	0.000284	7, 4	
	Vapor	NBT	0.000038	6, 15	
	Gas	NTP	0.000440	6, 15	
Viscosity μ Gm/cm-sec	Liquid	NBT	0.0001317	8	
	Vapor	NBT	0.0000109 ^g	3, 6, 16, 17	
	Gas	NTP		h	

Property Values of Parahydrogen at Selected Conditions					
Property			Value	Reference	
Molecular Weight			2.01594	1	
Triple Point Values	Temperature, °F		-434.83	2,9	
	Pressure, psia		1.02	9	
	Density, lb/ft ³	Solid			
		Liquid	4.81	2,9,14	
		Vapor	0.00784	9,14	
Normal Boiling Values	Temperature (T _b), °F		-423.187	2,9	
	Density, lb/ft ³	Liquid	4.42	2,9,14	
		Vapor	0.0835	9,14	
Critical Values	Temperature, °F		-400.30	9,14	
	Pressure, psia		187.6	9,14	
	Density, lb/ft ³		1.96	9,14	
One Gallon Liquid (NBT) Equivalents	Weight, lb		0.5907	9	
	Volume of gas, ft ³	NBT	7.07	2,9	
		NTP	113.3	2,6,9	
		Equivalent Volumes of Gas per Volume of Liquid (NBT)		NBT	52.9
		NTP	848	2,6,9	
Heat of Fusion, Btu/lb			TP	25.07	10
Heat of Vaporization, Btu/lb			NBT	191.7	9,10,14
Specific Heat Btu/lb-°F	C _s	Solid	TP	0.674	3,10
		Liquid	NBT	2.30	12
		Vapor	NBT		c
	C _v	Liquid	NBT	1.35	13,14
		Vapor	NBT	1.55	14
		Gas	NTP		d
	C _p	Liquid	NBT	2.3	14
		Vapor	NBT	2.91	14
		Gas	NTP		e
Specific Heat Ratio	C _p / C _v	Liquid	NBT	1.72	13,14
		Vapor	NBT	1.902	14
		Gas	NTP		f
Thermal Conductivity K Btu/hr-ft-°F		Liquid	NBT	0.0687	7,4
		Vapor	NBT	0.0092	6,15
		Gas	NTP	0.106	6,15
Viscosity μ Centipoise		Liquid	NBT	0.01317	8
		Vapor	NBT	0.00109	g
		Gas	NTP		h

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IV. RECOMMENDED MATERIALS AND PRACTICES FOR USE WITH LIQUID FLUORINE

- A Compilation from the Literature with Annotated Bibliography -

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BIBLIOGRAPHY

1. HAZARDS

1.1 Health

1.1.1 General - Liquid fluorine exhibits a slight green fluorescence giving it a yellow green appearance. Pure gaseous fluorine has a pale yellow color. A fluorine cloud released into the atmosphere generally has a dense, medium brown appearance. This changes rapidly to a milky-white appearance as the fluorine reacts with the water in the atmosphere to form hydrogen fluoride.

Fluorine has the characteristic halogen odor; however, under most circumstances, HF is formed by the time its odor can be detected, which adds a faint ozone odor.

1.1.2 Toxicity - Both liquid and gaseous fluorine are exceedingly corrosive to body tissues. The low boiling point of fluorine makes it unlikely that the liquid will get in contact with the body, but if this accident should occur, skin injury resembling burns will follow. These are likely to be severe, deep, and slow in healing.

Gaseous fluorine in high concentrations will result in thermal burns to the skin. This type of burn would be similar to that caused by an oxyacetylene flame and could be treated like any combination chemical and thermal burn. Gaseous fluorine in moderate concentrations in contact with the skin will result in burns more chemical in nature and closely resembling those caused by hydrofluoric acid. A latent period depending upon the degree of exposure is usually associated with fluorine burns. Several hours may elapse before the patient is conscious of pain or injury.

The lesion becomes first reddened and then swollen and pale with a macerated appearance. It is accompanied by severe throbbing pain. Adequate treatment will usually stop the pathological changes at this stage; otherwise necrosis and ulceration will ensue.

Because treatment of fluorine burns is specific and highly efficient, it should be instituted immediately, even in cases of questionable severity. The skin should be flushed with copious amount of tepid tap water for at least 15 minutes, the washing should not be interrupted even while removing contaminated clothing. Medical attention should be summoned and, if in the opinion of the physician the burn is mild, a water base paste containing magnesium salts may then be applied (A commercial paste on the market consisting of 20% magnesium sulfate, 6% magnesium oxide, 18% glycerin, 1.2% procaine hydrochloride, q.s. water base). If the physician considers even a slight chance that the burn may develop beyond the erythema stage, the tissue beneath and around the affected area should be infiltrated with 10% calcium gluconate. This precipitates the fluorine as inert calcium fluoride.

Fluorine burns of the eye require immediate, copious and prolonged irrigation with tepid tap water. Subsequent treatment should be directed by an ophthalmologist and consists in the application of pontocaine for the relief of pain, mydriatics, and the removal of any necrotic tissue in the cornea.

Under no conditions should the first aid treatment consist of the application of oil or ointments to either the skin or eye.

The effect of chronic long term exposure to gaseous fluorine may cause chronic pulmonary damage and deposition of fluoride in bones and teeth. Tests at Edwards AFB in which volunteers were subjected to concentrations of about 25 ppm for a period of even less than a minute showed the exposed person has a sore throat and minor chest pains for a period of six hours. Emergency tolerance limits (ETL) for fluorine are dependent on exposure time. The following tabulated values are for seldom or non-occupational frequency of exposure such as is likely to be encountered in rocket operations

Exposure Time in Minutes	ETL in ppmv
5	5
15	3
30	2
60	1

0.5 ppmv is the ETL for repeated exposure. 0.1 ppmv/8 hour day has been accepted for occupational exposure TLV (threshold limit value).

The inhalation of flood concentrations of fluorine would probably cause asphyxia by mechanism of laryngeal and bronchiole spasm, and later by bronchiole obstruction and pulmonary edema. Exposure to high concentrations of fluorine would also be accompanied by gastrointestinal symptoms and irritation of the eyes, throat and skin; but these symptoms would be secondary in importance to lung damage.

1.1.3 First Aid and Self Aid - Treatment of fluorine burns is specific and highly efficient and is discussed in the previous section. In the event of exposure of persons to concentrations of fluorine vapor exceeding the hygienic standards, remove the patients from the contaminated atmosphere and keep them as quiet as possible. If breathing stops, apply artificial respiration and get medical attention as soon as possible. If there is evidence of air hunger, oxygen therapy should be instituted. Administration of the gas under slightly increased pressure may force more oxygen past the stenosed bronchiole tubes, and an increased oxygen tension in the alveoli will result in more efficient transfer of oxygen to the blood. If the patient does not succumb to anoxemia within a few hours, complete recovery can be expected and residual conditions should be treated symptomatically.

1.2 Fire

1.2.1 General - Fluorine is the most powerful chemical oxidizing agent known; it reacts with practically all organic and inorganic substances, including water. The few exceptions are the inert gases, metal fluorides in their highest valence state, and a few (catalyst and adulterant free) completely fluorinated organic compounds. Even the latter may burn in a fluorine atmosphere if contaminated with a combustible material or if subjected to high flow velocities of fluorine. The heats of reaction are always high and most reactions take place with ignition.

1.2.2 Control - The reaction of fuels with liquid fluorine is frequently so rapid that no attempt can be made to ex-

tinguish them. After consumption or dissipation of the fluorine in the fluorine-fed fire, extinguishing methods appropriate to the nature of the resultant fire may be used. Small spills of fluorine may be handled by remote application of water fog to promote smooth rapid combustion of the fluorine. Areas surrounding large spills should be evacuated until the fluorine has evaporated.

1.3 Explosion

1.3.1 General - Fluorine itself is insensitive to explosive decomposition; however, explosive reactions may result in the action of fluorine on most materials. While fluorine "explosions" are not at detonation velocities the reaction of fluorine with other material is in some cases sufficiently violent that it may cause slight overpressures even in unconfined areas. In confined spaces such as tanks, lines, etc. the reaction of fluorine with some foreign material (e.g. condensed water) will initiate the reaction of fluorine with the transfer or storage container resulting in pressure ruptures and subsequent fires or secondary explosions. The hypergolicity of fluorine precludes the formation of detonable liquid mixtures, but explosive vapor mixtures may be formed with combustibles which can explode spontaneously.

Explosions may occur when liquid fluorine is trapped in a closed system and refrigeration is not maintained. Liquid fluorine trapped between valves, can cause violent rupture of the pipe or tube on warming. This condition is not unique with fluorine but it exists with all cryogenics.

1.3.2 Prevention - Any equipment to be used for fluorine service should be thoroughly cleaned, degreased and dried, then treated with increasing concentrations of fluorine gas so that any impurities may be reacted without the simultaneous ignition of the equipment.

2. SAFETY MEASURES

2.1 General - Although fluorine is the most reactive element and recognized as a very dangerous material, it can be handled without undue hazard if proper precautions are taken. It is important for the safe handling of fluorine that in addition to the safety measures given below and in subsequent sections that common sense safety precautions should not be overlooked. All operations involving the handling of fluorine shall be performed by persons working in a group. Trained supervision of all potentially hazardous activities involving fluorine is essential.

The NASA-Lewis Research Center policy¹ on use of protective clothing and equipment (breathing gear, etc.), which has proved effective in almost two decades of safe operation with fluorine systems, can be summed up in one sentence: Use protective equipment only when it improves safety. Extravagant use of protective clothing may provide a false sense of security, while in fact being only a physical hindrance. The following table gives various conditions and recommended clothing or apparatus to be used in each condition:

TABLE 1 - RECOMMENDED SAFETY CLOTHING AND EQUIPMENT

When the condition is, or is expected to be, one in which -	Wear the following equipment: ^a
There is no odor of fluorine (less than 0.10 ppm fluorine in air)	Common work clothes
Fluorine can be smelled, but does not irritate the nose (0.10 to 15 ppm fluorine in air)	Easily removable plastic gloves, face shield, cloth head covering, work clothes; a filtered - air mask, or a portable air supply system should be used whenever the odor of fluorine is detected and persists for longer than one minute
Fluorine irritates the nose, but does not affect the skin or hair (15 to 100 ppm fluorine in air)	Filtered-air mask or portable air supply, easily removable plastic gloves, face shield, cloth or plastic head covering, and loose-fitting plastic jacket; minimize time in the area
Fluorine warms the skin and makes body hair sticky (above 100 ppm fluorine in air)	Full safety suit (preferably made of a fluoropolymer) and breathing-air supply
Rescue, standby, or accident surveillance as needed	Full safety suit and breathing air supply

^aComfortable footgear does not exist which would give protection against fluorine splash, impingement, or cryogenic puddles. Common work shoes are considered appropriate protection for the feet. Routine precautions should be followed to prevent spilling any cryogenic fluid into pockets, on shoe tops, sleeves, etc.

¹ Table 1 and the accompanying three paragraphs have been excerpted with slight modification from NASA SP-3037, HANDLING AND USE OF FLUORINE AND FLUORINE-OXYGEN MIXTURES IN ROCKET SYSTEMS by W. H. Schmidt.

Remotely charged and operated facilities are manipulated by personnel in everyday work clothes, except for those involved in the connection and manual valve operation of the fluorine supply. This function requires only a face shield¹ and gloves (TFE cloth impregnated with TFE, or other fluorinated polymer²) for protection from fluorine "puffs" when removing tubing caps, or at other times of inadvertent exposure. High pressure gaseous systems that are manually operated must be provided with barriers and shielding for protection, which, in most cases, eliminates the need for additional protective clothing. All protective clothing should be designed for rapid and easy removal, in case a fluorine impingement should penetrate the body covering. When breaking into a system that has contained fluorine, personnel should wear

- 1) a transparent plastic full face shield¹ over a head covering of cloth or plastic,
- 2) gloves made of TFE cloth impregnated with TFE, or one of the other fluorinated polymers², designed for easy removal by vigorous arm movements,
- 3) a jacket, or similar covering, also made of TFE cloth impregnated with TFE, or one of the other fluorinated polymers², (also designed for ready removal).

If fumes cannot be avoided or if exposure is prolonged, filtered breathing air from a supply tank or a portable breathing apparatus, with self-contained supply tank and face mask, is required.

2.2 Personnel Education - The following subjects shall be explained thoroughly to all personnel concerned with the handling, transfer, and storage of fluorine:

- (a) Nature and properties of fluorine, with emphasis on its toxicity.
- (b) Approved materials which are compatible with fluorine.

- (c) Proper equipment and its operation.
- (d) Use and care of protective equipment and clothing.
- (e) Safety, self-aid, and first-aid instructions.

2.3 Personal Protection - The principal personal hazards associated with the handling of fluorine are as follows:

- (a) Inhalation of vapor.
- (b) Exposure of the body to the liquid or vapor.
- (c) Fire.

No materials are known at present that will provide complete protection. Impermeable gloves, boots, and body protection shall be worn when handling fluorine in all uncertain or highly hazardous activities. All such

¹ Face shields will not provide protection against a high-velocity impingement of fluorine. While amyl butyrate has been used for face shields, another material, Aclar, a film made from fluorohalocarbon, is preferred.

² While the TFE cloth is preferred, neoprene is also suitable for protection against inadvertent exposure to dilute puffs of fluorine.

protection clothing shall be designed and used in such a manner that it can be shed easily and quickly.

3. TRANSFER AND STORAGE

3.1 General - Liquid fluorine must be stored in containers (either fixed or mobile) of approved design, materials, and construction.

Storage, transfer, and test areas must be kept neat and free from combustibles. These areas must be inspected frequently.

In designing a reliable fluorine system the greatest emphasis is placed on three factors:

- (a) Achieving an absolutely leak proof system.
- (b) Avoiding irregularities in the flow passages such as crevices and lap joints, weld slag, rough or oxidized surfaces.
- (c) Taking extreme care in the assembly, cleaning, passivation, and in the prevention of contamination.

The success of this procedure has been demonstrated by many successful operations of liquid fluorine at the NASA Lewis Research Center. It should be mentioned here that much of the material presented in this section was derived from the paper "Some Problems in using Fluorine in Rocket Systems" by H. W. Schmidt and E. A. Rothenberg, as presented in O. S. U. Special Report No. 12 entitled PROCEEDINGS OF THE PROPELLANT THERMODYNAMICS AND HANDLING CONFERENCE - this is referenced in the General Bibliography of the present document. For a comprehensive, much expanded treatment of the subject of fluorine, the reader is referred to NASA SP-3037, HANDLING AND USE OF FLUORINE AND FLUORINE-OXYGEN MIXTURES IN ROCKET SYSTEMS by H. W. Schmidt - a report which is referenced in the fluorine bibliography following this section.

3.2 Materials - Partially oxidized material can be fully oxidized simply by exposure to an oxidizing atmosphere of suitable temperature. This is particularly true with fluorine since fluorine has the highest oxidation potential of all the elements. Ordinary oxides may be considered to be in a state of less than maximum oxidation because fluorine is capable of replacing the oxygen atoms with sufficient heat release to maintain combustion. Most metallic oxides react with fluorine at temperatures less than -250°C (-419°F). Even fire brick ($\text{Al}_2\text{O}_3 - \text{SiO}_2$) will burn in fluorine. The reaction of fluorocarbon polymers with fluorine is another example of a reaction of a higher state of oxidation. Polytetrafluoroethylene ($-\text{CF}_2-\text{CF}_2-$), for example, is a saturated fluorocarbon chain but fluorine is capable of breaking the carbon bond reacting the carbon to a higher degree of saturation forming carbon tetrafluoride. The reaction may be initiated from an unreacting system by increasing the temperature.

A pressure increase will also initiate reaction with fluorine. Several non-metallic materials were tested under the static conditions for compatibility with both liquid and gaseous fluorine at atmospheric pressure and at 1500 psig respectively. In a majority of the tests on which no reac-

tion occurred at atmospheric pressure, reaction was initiated by the pressure increase.

Metallic materials with a high thermal conductivity resist ignition with fluorine more readily than materials with low conductivity. Combustion with fluorine will not occur if the heat of reaction can be dissipated from the point of ignition fast enough to maintain less than the combustion temperature for the material involved.

Fluorocarbon materials undergo two types of reaction with the fluorine--a slow reaction in which the material slowly reacts forming fluoride at the surface at a relatively low temperature, and a fast combustion reaction. The type of reaction is generally determined by the ability or the capacity of material adjacent to the reacting zone to dissipate the heat of reaction at a rate equal to or greater than the rate in which the heat is produced. Fluorocarbon O-rings or valve stem packings closely surrounded by metal slowly react away if the gaseous fluorine is able to leak past them. However, active combustion is generally prevented because of the high thermal conductivity of the system surrounding the O-ring or packing.

Fluorine will react with the surface of nearly all solid materials. If the material is spontaneously combustible, reaction will continue until the fluorine or the material is depleted. If the material is not spontaneously combustible, like most metals, surface reaction simply forms a fluoride film on the surface. This film, if sufficiently tenacious, is considered generally to be an aid in preventing fluorine attack on fluorine systems. If, however, the surface area exposed to fluorine is very large in proportion to the mass, for example, a fine mesh screen or finely divided material such as powdered metal or spun glass, the surface reaction will initiate combustion and the material becomes spontaneously reactive.

Many fluorine system failures occur during flow conditions, although the flow condition itself has not been established as the direct cause of failure in most cases. In one test at NASA Lewis, polytetrafluoroethylene (Teflon) was exposed statically to liquid fluorine at 1500 psi without reaction. The same material when subjected to flow conditions at 50 psi reacted violently. The fact that Teflon withstood static exposure to liquid fluorine and yet failed in the dynamic test is of particular interest. Metals form a protective fluoride surface film when exposed to fluorine. Teflon, on the other hand, tends to react with fluorine to break down the polymer and form unsaturated low molecular weight fluorocarbons. These fluorocarbons would not adhere to the surface and therefore would be of no value as protective films. It is also possible, of course, that the surface impurity could act as a reaction initiator although this should be independent of kinetic or static conditions.

In the experience at NASA Lewis in fluorine system failures, combustion reaction was initiated with fluorine and its containing system. The section in which failure occurred was usually consumed which destroyed the evidence so that the exact cause could not be determined. However, high pressure flow of liquid fluorine was most frequently involved

and the compatibility of materials was suspected. A series of compatibility tests with liquid fluorine were conducted in an effort to isolate the cause of burnouts. Flow tests were run at pressures up to 1500 psi. Fluorine was cycled to the test sections by means of an appropriate control valve in pressures up to 1500 psi. The materials tested were Monel, stainless steel, nickel, brass, and aluminum. Pinhole orifices were used to simulate leaks for determining erosion tendencies. Fluorine was impinged against flat-ended plugs at velocities over 350 ft. per sec. and sharpened wedges were used to study turbulence and the effect of sharp edges in fluorine flow. Liquid fluorine at 1500 psi was released suddenly into one-quarter inch stainless steel and aluminum tubing containing gaseous fluorine at ambient temperatures without adverse effect on the system. No metal erosion or appreciable chemical attack occurred on any of the metal specimens used and tested. Pressure, flow rate, Reynolds number, or system configuration had no effect on the test. After the entire preparation and operating procedure was reviewed it was concluded that the primary cause of burnouts in fluorine systems was not associated with the selection of materials or configuration of system components, but rather must be due to system contamination. Contamination includes all foreign material, organic or inorganic that is not fully fluorinated. Use of extreme care in assembly, together with astringent cleaning procedures to obtain meticulously clean systems resulted in a marked reduction in system failures.

A secondary cause of "burnouts" may be attributed to mechanical failure of the containing system which may allow the fluorine to come into contact with reactable material. For example, a flange leak may allow fluorine to contact moisture or dirt on the outside of the flow line which may initiate combustion with the type material.

Most metal materials of construction are resistant to fluorine attack if the conditions of exposure are not too severe. In many cases, this resistance is enhanced by the fluoride film that forms on or in the surface of the material from initial reaction. Materials more subject to fluorine and hydrogen fluoride corrosion accumulate relatively heavy fluoride coatings during prolonged use. Iron is such a material and is used mostly in gaseous fluorine systems at low pressures in moderate ambient temperatures. The advantage is low cost; the disadvantage is flow restriction and clogging from accumulated solids which necessitates more frequent maintenance. Fluoride coatings are sometimes very brittle, sometimes porous and powdery. The more resistant materials such as nickel and stainless steel rarely exhibit visible surface films from fluorine exposures. The permeability of the fluoride coating, which varies with the material, is one of the controlling factors in the corrosion rate. Those metals with greatest resistivity to fluorine attack are least dependent on the fluoride film for protection. For example, nickel is nearly passive to liquid fluorine, and may be exposed to severe dynamic conditions without prior surface passivation. Other commonly used materials with excellent resistivity to fluorine are Monel, Inconel, 300 series stainless steel, brass, copper, and aluminum.

There are no non-metals that are known to be entirely

resistant to reaction with liquid fluorine. One reference reports that the ceramic material mullite was not visibly affected by the liquid fluorine, but it is reasonable to assume that if enough heat was generated to melt or burn most metals, the fluorine could attack the ceramic material.

Fluorine reacts with organic, aqueous, or siliceous materials otherwise considered inert, as well as with oxidizable materials. Therefore, silicants and standard petroleum-based lubricants are not usable. There are no reliable lubricants for fluorine service.

3.3 Equipment - Liquid fluorine handling equipment shall be degreased by washing with approved grease-removing solvents before being used. Passivation is recommended as a final treatment prior to liquid exposure. Equipment taken out of service for maintenance or modification shall be inspected and cleaned before returned to service. The tanks for storage of liquid fluorine are usually constructed with three horizontal concentric shells; an outer shell, an intermediate shell and an inner shell. As dictated by the user, the shells can be constructed of either Monel, aluminum, or stainless steel. The inner shell will contain liquid fluorine; the intermediate shell will contain liquid nitrogen; and the outer shell an insulating material of low thermal conductivity such as Santocel or perlite provided there is no concern of potential fluorine leakage into the insulating annulus. In the event there is a probability that fluorine may seep into the insulated space, it is recommended that a vacuum only, or a high vacuum plus a metallic radiation shield, be utilized. The primary objective in fluorine tank fabrication is to achieve a smooth crevice free interior; seam welds which have flux and slag inclusions, pockets or bubbles, and oxidized surface flaking are considered particularly hazardous, and this type of contaminant is capable of reacting and combusting with fluorine. The higher the state of purity of the container, its surface and even the fluorine itself, the less possible it is for reaction to occur. It follows that pressurizing gases must be free of moisture or other contaminants.

3.3.1 Containers - Liquid fluorine may be stored in either fixed or mobile tanks of approved design and materials. Storage and shipping containers designed for non-cryogenic fluids shall not be used in this service. Storage tanks shall be proof-tested prior to service in accordance with the provisions of applicable ASME, ASTM, or ICC specifications for pressure vessels. Containers for shipment, storage and transfer of liquid fluorine shall be fabricated in accordance with the physical and structural requirements dictated by the use for which they are intended.

3.3.2 Lines and Fittings - For lines and fittings the objective again must be a leakproof plainly designed system free of contamination. A reliable way to join fluorine flow lines is to arc weld them in an inert helium atmosphere (Heliarc process) using a V-notch butt weld; for removable sections, the use of concentrically serrated flanges bolted together against an annealed soft aluminum gasket is acceptable. Bolt tension should be checked frequently, especially after several extreme temperature cycles.

Compression fittings and threaded connections have been used successfully for high pressure fluorine systems in lines

less than 3/4 in. diameter, but for 3/4 in. or above they are not considered a reliable joint for liquid fluorine service. Compression fittings above 3/4 in. are more difficult to seat properly than the smaller sizes, thereby increasing the possibility of high pressure leaks. Also they tend to fail more readily from high pressure by tubing pulling out of the fittings.

The threaded type connections should be avoided but have been used successfully when the threaded portion has been silver soldered in place or better if both ends of the threaded section have been welded or brazed; this prevents fluorine from getting into the spaces between the threads where cleaning is difficult.

3.3.3 Valves - Many of the standard globe, plug or needle valves can be adapted for gaseous fluorine by using Teflon valve stem packings. In most cases, liquid fluorine requires that diaphragm- or bellows-sealed valves be used to prevent liquid from contacting the seal material. Because of the extremely low temperatures of liquid fluorine systems, standard valves packed with Teflon or Kel-F are unsuitable although some reliability can be achieved by using a valve on an extension 10 to 12 inches long; vertically mounted valve-bonnet extensions reduce the possibility of liquid fluorine contacting the stem packing since the heat transfer into the valve maintains a gas pocket in the area of the packing. This provides higher packing temperatures, thus preventing leaks that may cause spontaneous ignition.

Hand-operated valves should be equipped with valve stem extension through a suitable barrier for the protection of the operator. This type of operation should be limited to low pressure (<500 psi) gaseous systems.

For high pressure liquid fluorine systems, remotely operated diaphragm- or bellows-type valves are preferred. Excellent results have been obtained with valve plugs of Monel or stainless steel and seats of nickel, brass, copper, or aluminum. Valves should be completely disassembled for thorough cleaning prior to use and while the valve is apart for cleaning it is good practice to mate the valve plug in its respective seat for perfect seating. Valve actuators may be either hydraulic or pneumatic.

3.3.4 Pumps and Hoses - The requirement for flexible tubing for liquid fluorine use is that it be all metal. Bellows-section or diaphragm flexer units are designed for a minimum of stress and strain of the required motion and should be adequate for most requirements. Difficulty could be expected if excessive surface flexing causes cracks, flaking or leaks in the tubing or if the design prevents suitable cleaning.

In the event a pump is utilized in a transfer operation, extreme caution should be exercised on the design of the shaft seal. Depending on the use, the seal should be designed to prevent fluorine leakage entirely, or to control the leakage and its path so as to prevent it from mixing or associating with other incompatible elements of the pump.

3.4 Transfer Procedures - Transfer of liquid fluorine is generally accomplished by pressurization with helium or by evaporation of the liquid. It is essential that all of the equipment, lines, and fittings be leak-tight, dry and thoroughly cleansed of all foreign material prior to use.

Because of the fact that fluorine is the most powerful oxidizing agent known, reacting with practically all organic and inorganic substances - exceptions being the inert gases, metal fluorides in the highest valence state, and a few (catalyst and adulterant free) completely fluorinated organic compounds - limited information can be provided here regarding transfer methods and techniques. The necessary procedures for transferring to and from storage containers and various items of equipment shall be based on specific component design features and specific requirements of each test, experiment, or application. All operating personnel shall have complete and thorough instruction prior to any transfer operation.

USE CAUTION --- DOUBLE-CHECK EVERY OPERATION

3.5 Spills, Leaks and Decontamination - All areas containing fluorine under pressure shall be inspected for leaks at suitable intervals. Do not move leaking fluorine cylinders. A leak in the cylinder wall is usually caused by an impurity in the cylinder and is often associated with a pressure rise which may cause the cylinder to rupture. When a valve is found to be leaking, no attempt should be made to tighten the valve or the packing nut. Evacuate the area.

Repair all leaks only after venting the fluorine. The use of filter paper moistened with potassium - iodide solution or moist starch-potassium iodide paper is a very sensitive means of detecting fluorine (down to about 25 ppm). The odor of fluorine is so strong that very low concentrations can be detected, but this is not a safe or reliable method of detecting lethal quantities.

Ammonium hydroxide (aqua ammonia) in a plastic squirt bottle should be used as a rapid qualitative test. This method is used for the detection and location of small gas leaks; the chemical smoke produced is very visible.

Small amounts of fluorine may be disposed of by slow venting. It may also be disposed of by a fluorine-hydrocarbon-air burner, scrubber, and stack. Possibly the safest disposal method is that in which fluorine is converted to unreactive and nontoxic carbon tetrafluoride by passing it through charcoal.

In the event that liquid fluorine is spilled, the contaminated area can be neutralized with sodium carbonate. The dry powder can be sprayed on to the spill area from a fluidized system similar in principle to that of dry chemical fire extinguishers. If major spillages occur in areas where formation of hydrofluoric acid liquid and vapor pose no undue danger, water in the form of a fine mist or fog is recommended. A major portion of the fluorine will be converted to hot, light gaseous products which rise vertically and diffuse quickly into the atmosphere.

3.6 Cleaning Procedures - Equipment is to be cleaned in accordance with the provisions of Appendix A.

3.7 Passivation Procedures - Passivation, of either system components or an assembled system, is the final step of cleaning for fluorine handling equipment. Prime importance was originally given to the fact that the procedure resulted in formation of a fluoride film which covered all

exposed (to fluorine) surfaces, thereby inhibiting any further corrosion and material surface reactivity in actual service. This is still considered to be one function of the procedure; however, its main benefit is in the complete fluorination of residual contaminants remaining in the component or system after normal cleaning procedures have been followed (see Section 3.6 above). There is no evidence at hand today that requires passivation for materials which have been thoroughly cleaned. This is not meant to indicate that passivation may be regarded as a substitute for good cleaning practices - it isn't. However, most system failures that have resulted in burnout have been traced to some form of contamination, and passivation provides one additional bit of insurance that everything has been done that can be done to assure extreme cleanliness where it is such an important factor. Specifically, the procedures to be used involve either of the following sequences:

A. Passivation by evacuation and pressurization

- 1) Evacuate system and backfill with pure, dry nitrogen or helium gas;
- 2) Evacuate system again and backfill with pure fluorine gas or a mixture of fluorine and helium gases until a slight

positive gage pressure is reached;

3) Hold for several minutes to permit any reaction that might take place to occur slowly;

4) Continue pressurization (with incremental stops to allow any reaction with contaminants to proceed slowly) until 110 percent of maximum operating pressure is reached;

5) Hold for at least 1/2 hour;

6) Vent system to slight positive gage pressure to prevent infiltration of atmospheric contaminants.

B. Passivation by pressurization without evacuation

1) Pressurize system to maximum operating pressure, or 50 psi, as desired with pure, dry nitrogen or helium gas;

2) Vent system to slight positive gage pressure;

3) Pressurize system with pure fluorine gas or a mixture of fluorine and helium gases (with incremental stops to allow any reaction with contaminants to proceed slowly) until 110 percent of maximum operating pressure is reached;

4) Hold for at least 1/2 hour;

5) Vent system to slight positive gage pressure to prevent infiltration of atmospheric contaminants.

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DESIGN HANDBOOK FOR LIQUID FLUORINE GROUND HANDLING EQUIPMENT (Second Edition) - Aerojet-General Corp., AFRPL-TR-65-133 (Aug. 1965) Contr. AF 04(611)-10541, 484 pp 126 fig 72 tab 265 ref - DDC AD 468 216.

This handbook is a compilation of available design data and methods of operation representing present technical experience levels as applied to fluorine. In addition, it includes design and operational data for liquid helium, liquid oxygen and liquid nitrogen systems in those areas likely to be of use to a designer of a liquid fluorine facility.

The handbook also contains considerable design information to assist the designer in scaling up current sizes of fluorine and cryogenic systems to meet the designer's specific requirements.

Because no exact system requirements are specified, no optimum system is selected, though several fluorine systems are discussed. Both advantages and disadvantages of the various components are discussed to enable the designer to choose the best system for his particular needs. Bases for comparison include cost, availability, length of operation, fluorine loss and location.

THE PROPERTIES AND HANDLING OF FLUORINE - Air Products & Chemicals, Inc., ASD-TDR-62-273 (Oct. 1963) Contr. AF 33(616)-6515, 131 pp 11 fig 57 tab 187 ref - DDC AD 423 751.

This report presents data on properties of fluorine, methods of analysis, safe-handling procedures, compatibility of materials of construction with fluorine, corrosion rates of metals, and bibliography of pertinent references.

FLUORINE (Product Information Technical Bulletins) - General Chemical Division, Allied Chemical and Dye Corporation, New York.

Information from "the nation's primary supplier of fluorine, in both gaseous and liquid forms for commercial and research applications" on the storage, handling, safety, and material compatibility of fluorine.

BIBLIOGRAPHY OF FLUORINE - Briggs, C. (Ed.), Martin Company, Denver, Colo. (July 1, 1961) 12 pp DDC AD 293 821.

This compilation includes titles of approximately 142 documents related to the use of fluorine in liquid rocket engines. Covered in this listing are books, reports, journals, and magazines prior to, and including, the year 1961.

BIBLIOGRAPHY OF FLUORINE AND FLUORINE-OXYGEN OXIDIZERS FOR SPACE APPLICATIONS - Cabaniss, J. H. - NASA TM X-53149 (Oct. 1964) 76 pp 353 ref.

The bibliography references approximately 350 reports on fluorine and fluorine-oxygen mixtures (FLOX). In the introduction, current government contracts pertaining to FLOX and fluorine are listed. The bibliography includes separate sections dealing with material compatibility; handling, storage, disposal, and safety considerations; physical and chemical properties; propellant oxidizer studies; vehicle component design studies; and miscellaneous reports.

A LITERATURE SURVEY OF THE CORROSION OF METAL ALLOYS IN LIQUID AND GASEOUS FLUORINE - Cabaniss, J. H. and Williamson, J. G. - NASA-George C. Marshall

Space Flight Center, Huntsville, Ala. - Internal Report MTP-P & VE-M-63-21 (Dec. 31, 1963).

A literature survey on the corrosive nature of both liquid and gaseous fluorine is presented. This paper contains general information regarding: (1) chemical reaction of fluorine with various metallic materials; (2) conditions under which these materials can be used with fluorine; (3) results of corrosion tests that have been conducted on various materials over the range of -320°F (-196°C) to 1300°F (704°C).

FLUORINE SYSTEMS HANDBOOK - Douglas Missile and Space Systems Division - NASA CR-72064 (July 1967) NASA Contr. No. NAS_W - 1351 413 pp 76 fig 54 tab 325 ref.

This Handbook contains criteria for the design of airborne fluorine feed systems and associated components. Two types of information are presented: (1) philosophical information defining general methods, and (2) detailed specifications and procedures. Although the major emphasis has been on criteria for components exposed to elemental fluorine, the information is generally applicable to systems utilizing other cryogenic oxidizers which contain fluorine as a constituent.

DEVELOPMENT AND DEMONSTRATION OF CRITERIA FOR LIQUID FLUORINE FEED SYSTEM COMPONENTS - Final Report - Douglas Missile and Space Systems Division - NASA CR-72063 (Oct. 1967) NASA Contr. No. NAS_W - 1351 - 473 pp 198 fig 67 tab 289 ref.

The objectives of the work covered by this report were: (1) to establish the criteria for use in the design, fabrication, inspection, and servicing of fluorine flightweight propellant feed systems, and (2) to demonstrate, where necessary, the adequacy of these criteria.

FLUORINE PROPULSION TECHNOLOGY - Flanagan, J. R. and Stephenson, F. W., Jr. - AIAA Second Annual Meeting, July 26-29, 1965, AIAA Paper No. 65-536.

This paper presents a review of the technological investigations that have been conducted to advance the state-of-the-art of fluorine propulsion. It also reports the key accomplishments in the current technological investigations and discusses the role that fluorine may play in our future space program.

A FACILITY FOR TESTING ROCKET ENGINES AND FLOW SYSTEM COMPONENTS IN LIQUID FLUORINE - French, J. R. and Robinett, F. E. - SAE Paper No. 670590 (June 1967) 6 pp 3 fig. [Published in Proc. of the SAE Aerospace Systems Conference, Los Angeles, Calif.].

A liquid fluorine closed flow loop and engine test position has been designed, constructed, and operated for extended periods with very few difficulties. This paper describes the design philosophy of the system and discusses the operation experience gained in rocket engine firings and cold flow operations. Based upon experience with this system, a new attitude toward the handling of fluorine has evolved. Fluorine may now be handled with confidence so long as its peculiar characteristics are considered and respected. In this light, more realistic safety, cleaning, passivation, and testing procedures have been developed and are described herein.

HANDLING FLUORINE AND FLUORINE COMPOUNDS - The International Nickel Company, Inc., New York.

A general information document concerning fluorine and fluorine compounds.

EXPERIMENTAL EVALUATION OF LIQUID FLUORINE SYSTEM COMPONENTS - DeWitt, R. L. and Schmidt, H. W. - NASA-Lewis Research Center, Cleveland, Ohio - NASA Tech. Note D-1727 (June 1963).

The investigation reported here was undertaken primarily to design, develop, and test prototype flight hardware to be used in systems for the safe ground-to-vehicle transfer of liquid fluorine and secondarily to test the capability of liquid fluorine with the materials and fabrication techniques used in construction of the hardware. The prototype hardware consisted of a "quick-disconnect" coupling and a valve, which were designed and fabricated at the Lewis Research Center, and a commercially procured rotating-vane flowmeter.

FRICITION, WEAR, AND DYNAMIC SEAL STUDIES IN LIQUID FLUORINE AND LIQUID OXYGEN - Hady, W. F., Allen, G. P., Sliney, H. E., and Johnson, R. L. - NASA-Lewis Research Center, Cleveland, Ohio - NASA Tech. Note D-2453 (Aug. 1964) 15 pp.

Friction and wear of four material combinations were determined running submerged in liquid oxygen and in liquid fluorine. Results of these studies and two face contact seals run submerged in liquid fluorine indicated that Al_2O_3 sliding against either a fused fluoride film on Al_2O_3 or a nickel-bonded TiC cermet are acceptable material combinations for fluorine seal applications. The presence of a fluoride film, either as an applied fused coating ($\text{CaF}_2 + \text{LiF} + \text{NiF}_2$) or as a film formed during sliding (NiF_2 on a nickel-bonded TiC cermet or possibly AlF_3 on Al_2O_3) in liquid fluorine was beneficial in reducing friction and wear of the Al_2O_3 riders.

HANDLING LIQUID FLUORINE IN ROCKET APPLICATIONS - Kimball, A. R., Advances in Cryogenic Engineering 5, 77-84 (Proc. of 1959 Cryogenic Eng. Conf.) Plenum Press, Inc., New York (1960) Paper B-1, 3 fig 1 tab.

Fluorine is extremely attractive as the oxidizer in rocket propulsion systems in view of the 40% performance increase that may be realized by replacing present day operational propellants with a high-energy combination. Development of fluorine propulsion systems has, however, been approached cautiously because of concern expressed in view of its extreme reactivity and toxicity.

In this paper a review is made of the present state-of-the-art (1959) and Bell Aircraft Corporation's findings in the areas of material compatibility, equipment fabrication, operating techniques, instrumentation, and safety when using liquid fluorine.

LIQUID FLUORINE - PRODUCTION AND HANDLING - Neumark, H. R. - Allied Chemical Corporation - Presented at 2nd Missile Liquid Propellant Symposium (Nov. 1964).

A discussion and clarification of some of the problems related to the use of liquid fluorine.

THE COMPATIBILITY OF VARIOUS METALS WITH LIQUID FLUORINE - Kleinberg, S. and Tompkins, J. F. - Air Products, Inc., Allentown, Pa. - ASD-TDR-62-250 Report for Oct. 1960-Oct. 1961 on Finishes and Materials Preservation (Mar. 1962) Contr. AF 33(616)6515, 114 pp 22 fig 19 tab 14 ref.

Studies were made of liquid fluorine and its contaminants, reactions on metal surfaces, fluoride films, and immersion tests of tensile specimens. A method of preparing contaminant-free fluorine and its infrared analysis is discussed.

COMPATIBILITY OF POLYMERIC MATERIALS WITH FLUORINE AND FLUORINE-OXYGEN MIXTURES - Russell, L. M., Schmidt, H. W., and Gordon, L. H. NASA-Lewis Research Center, Cleveland, Ohio - NASA Tech. Note D-3392 (June 1966) 39 pp 17 fig 5 tab 3 ref.

Compatibility tests were performed on a number of polymeric materials with the use of various mixtures of fluorine and oxygen in both gaseous and liquid states. The purpose of these tests was to investigate the feasibility of using fluorine-oxygen mixtures in rocket-propulsion systems containing some nonmetallic materials. The tests were divided into two major areas, static tests and dynamic tests.

In the static tests, a number of test samples were exposed to various FLOX (fluorine-oxygen) mixtures, both gaseous and liquid, at atmospheric pressure and virtually static conditions in order to obtain information on compatibility solely as a function of fluorine concentration. The reactivity of the materials tested with FLOX under static conditions is a function of the concentration of fluorine in the mixture.

In the dynamic tests, selected materials were exposed to fluorine and FLOX at various combinations of concentration and flow velocity. Reactivity profiles were generated for these materials as functions of these two parameters. At any given fluorine concentration, flow velocity was a strongly significant parameter in the reactivity of FLOX with all materials tested. Generally the fluorocarbon polymers, particularly the fully fluorinated, straight-chain polymers were the most compatible with fluorine and with FLOX.

In both static and dynamic tests, a comparison between cryogenic liquid and ambient-temperature gaseous test results indicated that the liquid was the more reactive.

It was concluded that some of the materials tested may be considered for use in rocket systems with fluorine or FLOX under controlled conditions of exposure; however, because of possible variations in quality and because polymers are more sensitive to contamination than metals in a fluorine environment, they should be used with a margin of safety.

COMPATIBILITY OF METALS WITH LIQUID FLUORINE AT HIGH PRESSURES AND FLOW VELOCITIES - Schmidt, H. W. - NACA - Lewis Flight Propulsion Laboratory, Cleveland, Ohio - NACA Research Memo. E58D11 (July 1958) 15 pp.

Nickel, stainless steel, brass, and aluminum were evaluated for compatibility with liquid fluorine at flow velocities

up to approximately 400 ft/sec and pressures up to 1500 lb/sq. in. gage. Configurations were varied to produce severe turbulence and impact effects. Two rotating-vane flowmeters were also tested. None of the metals exhibited any measurable physical or chemical changes. In a run made with Teflon, fluorine reacted violently.

HANDLING AND USE OF FLUORINE AND FLUORINE - OXYGEN MIXTURES IN ROCKET SYSTEMS - Schmidt, H. W. (with assistance of J. T. Harper, Technical Writer) - NASA-Lewis Research Center, Cleveland, Ohio - NASA SP-3037 (1967) 279 pp 76 fig 42 tab 259 ref.

The accumulated technology necessary for the practical application of fluorine as a rocket propellant is presented in this report. Physical and chemical characteristics peculiar to fluorine are considered in relation to specific areas in design and development of rocket systems and in testing and launch operations. The information given herein should provide the designer, the engineer, or the scientist with information peculiar to the characteristics of fluorine and fluorine-oxygen mixtures (FLOX) to enable him to work with these fluids intelligently and safely. An effort has been made to present practical information for facility and systems design, assembly, and operation that is directly applicable to specific fluorine or FLOX programs.

PRODUCTION, HANDLING AND SHIPPING OF ELEMENTAL FLUORINE - Siegmund, J. M. - Allied Chemical Corporation, Industrial Chemicals Division, Morristown, N. J. - Paper No. 26a presented at AIChE National Meeting, Houston, Texas (Feb. 19-23, 1967) 6 pp 9 fig 1 ref.

The manufacture of fluorine on a large commercial scale is discussed relative to current production status, expansion capability and raw material availability. The transportation of liquid fluorine is discussed in detail. Construction materials suitable for various system components, and procedures for preparing the components for use with fluorine, are given. The toxic effects of fluorine, both chronic and acute, are discussed. Exposure probabilities in the aerospace industry are compared with those in the chemical industry. The need for different toxicity guidelines for each is discussed. Methods of detection and decontamination in the event of leakage or spillage are also given.

SOME FIELD PROBLEMS ASSOCIATED WITH THE HANDLING OF FLUORINE AND FLOX - Siegmund, J. M. - Cryogenic Technology 2, 105-108 (Sept./Oct. 1966).

Possible solutions to some of the problems associated with the handling of fluorine and FLOX mixtures in missile applications are discussed. FLOX is a physical mixture, not a chemical compound. Because of the difference in boiling points of the two components, a composition change will take place with FLOX if the oxidizer tank is vented to atmosphere during "holds" on the launch pad. The amount of change is shown in a plot of % boil-off versus composition. In order to accomplish the desired mission, it will probably

be necessary to build up the fluorine and oxygen content to the original composition prior to "lift off." A practical method for analyzing the FLOX oxidizer quickly, to determine fluorine content, is described. A method for making corrections for "boil-off" using a "boil-off" composition chart is presented, and an alternate method preventing "boil-off" is proposed.

When working with high energy toxic materials, it is necessary to monitor surrounding areas to maintain toxicant concentrations within tolerable limits. This paper discusses some experiences with an instrument for the detection of small amounts of fluorine in the atmosphere.

Quantity-distance relationships, fire, explosion, blast effects and medical aspects are discussed. The method of charging flox into 5,000 pound transport tanks is outlined.

PREPARATION, PROPERTIES, AND TECHNOLOGY OF FLUORINE AND ORGANIC FLUORO COMPOUNDS - Slessor, C. and Schram, S. R. (Ed.) - Div. VII, Vol. I, National Nuclear Energy Series, McGraw-Hill Book Company, Inc., New York (1959).

CORROSION OF METALS BY LIQUID FLUORINE - Singleton, A. H., Tompkins, J. F., Kleinberg, S., and Sterner, C. J. - Ind. & Eng. Chem., 57 (March 1965) pp 47-53.

In this study, metals were exposed to corrosive action of liquid fluorine for periods up to one year in duration. The metals tested were those most commonly of interest for this service - alloys of aluminum, titanium, copper, magnesium, nickel, and stainless steel. Samples were exposed in both the stressed and the unstressed states, and tensile strength of certain metals was tested after fluorine exposure. In all cases, it was concluded that the corrosive action of fluorine in a dry system, free from contaminants, is negligible, and that stress corrosion and cracking are not likely to occur in metals exposed to liquid fluorine. All metals tested had the same yield strength after exposure as before.

THE COMPATIBILITY OF VARIOUS METALS AND CARBON WITH LIQUID FLUORINE - Sterner, C. J. and Singleton, A. H. - Air Products, Inc., Allentown, Pa. - WADD TR 60-436, Report for June 1959 - June 1960 on Finishes and Materials Preservation (Aug. 1960) Contr. AF 33(616) 6515 110 pp fig tab 162 ref - DDC AD 244 309.

Investigation was made of the compatibility and resistance to corrosion of various alloys of Al, stainless steels (including high strength steels), Ti, Cu, Ni, Mg and Monel metal. Tests included continuous immersion in liquid F for periods up to 2 weeks; impact sensitivity (impact ignition of Ti and Al in liquid F and of Ti in liquid O at impact energy levels ranging from 2.6 to 65 ft-lb, and impact on tubes containing liquid F); passivation and storage for periods up to 64 days followed by immersion in liquid F; thermal shock of samples by liquid F and in contact with liquid F; flexing

and tearing of metal samples while immersed in liquid F; explosibility of contaminant in liquid F. Corrosion of metals in pure liquid F was negligible, generally amounting to less than 1 mil penetration per year. The results indicated that contamination of liquid F could result in severe corrosion. Graphite C was incompatible with liquid F, while dense amorphous C was only slightly affected. Ti ignited under impact in liquid F, but the ignition did not propagate. No evidence was found to support the theory that a F film is required to protect metals from attack by liquid F. Passivation by gaseous F was recommended as an extension of the cleaning procedure, although there is no evidence that passivation is required for materials which have been thoroughly cleaned.

THE COMPATIBILITY OF VARIOUS METALS WITH LIQUID FLUORINE - Sterner, C. J. and Singleton, A. H. - Air Products, Inc., Allentown, Pa. - WADD TR 60-819 Report for June 1960 - Oct. 1960 on Finishes and Materials Preservation (Mar. 1961) Contr. AF 33(616)6515 42 pp tab 15 ref - DDC AD 260 087.

Studies were made to determine the compatibility and resistance to corrosion of various metals with liquid F at -320F. Metals tested included various alloys of Al, stainless and high-strength steel, Ti, Cu, Monel, Ni, and Mg. It was found that the total corrosion of Ti which had been exposed to liquid F was independent of time for periods up to 2 weeks. The corrosion of Mg increased up to one day, but longer exposures up to 2 weeks produced no further corrosion. Increased corrosion of metals by F contaminated with water does not occur at liquid F temperatures, but takes place in the gas phase while the system is warming. F which had been liquefied in glass cells was found to contain solid material. Pretreatment with F gas is recommended since it can burn off surface contamination which might prove dangerous during liquid exposure. Abrading metal surfaces under liquid F with wire bristles caused no apparent increase in corrosion rate.

COMPATIBILITY PROBLEMS WITH MATERIALS OF CONSTRUCTION IN LAUNCH VEHICLES - The Boeing Company, Launch Systems Branch, Aero-Space Division, Huntsville, Ala. (March 1966) - Document D5-13229 16 pp 7 fig 1 tab 6 ref.

This report is a brief review of previous studies on the compatibility of various materials with LOX and FLOX mixtures that was presented at the 5th Launch Systems Branch Technology Council Meeting, May 26, 1965 in New Orleans, Louisiana. It is being released as a supplement to SAMRM-19 as a background reference on compatibility problems related to material exposed to oxygen and fluorine oxidizers in propulsion systems.

PHYSICAL PROPERTIES OF FLUORINE

PROPERTY VALUES OF FLUORINE
AT SELECTED CONDITIONS

Nomenclature and Conditions

TP = Triple Point

NBT = Normal Boiling Temperature (and 1 atm.)

NTP = Normal Temperature and Pressure
(70°F, 14.7 psia)

273.15°K* = 0°C = 32°F = 491.67°R

The term "mole" as used here means "gm-mole."

a. Taken from reference 5 at a pressure of one atmosphere and at a temperature of 85.24°K, which is not the best value of Normal Boiling Temperature (85.03°K) reported in the present work.

b. Calculated from $PV = RT + BP$, with B from reference 3.

c. Extrapolated value.

*Changes have been made to correct data to this scale where necessary.

Property Values of Fluorine at Selected Conditions				
Property		Value	Reference	
Molecular Weight		37.9968	1	
Triple Point Values	Temperature, °K	53.54	2	
	Pressure, mm Hg	1.66	2	
	Density, mole/cc	Solid 0.0500	3	
		Liquid 0.04487	3	
		Vapor		
Normal Boiling Values	Temperature (T _b), °K	85.03	2, 3	
	Density, mole/cc	Liquid 0.03966	4	
		Vapor 0.0001483 ^a	5	
Critical Values	Temperature, °K	144	6	
	Pressure, mm Hg	41800	6	
	Density, mole/cc	0.01241	5	
One Liter Liquid (NBT) Equivalents	Weight, kg	1.507	4	
	Volume of Gas, liters	NBT 265.78	5	
		NTP 956.83	3	
Equivalent Volumes of Gas per Volume of Liquid (NBT)	NBT 265.78		3, 4	
	NTP 956.83		7, 4	
Heat of Fusion, Cal/mole		TP 121.98	2, 3	
Heat of Vaporization, Cal/mole		NBT 1561.3	2, 3	
Specific Heat Cal/mole-°K	C _p	Solid TP 12.210	2	
		Liquid NBT		
		Vapor NBT		
	C _v	Liquid NBT		
		Vapor NBT		
		Gas NTP		
Specific Heat Ratio	C _p /C _v	Liquid NBT 13.948	2	
		Vapor NBT		
		Gas NTP		
Thermal Conductivity K Cal/cm-sec-°K	μ	Liquid NBT 0.000378	8	
		Vapor NBT 0.0000172 ^c	9, 10	
		Gas NTP 0.0000633	9, 10	
Viscosity μ Gm/cm-sec	μ	Liquid NBT 0.00243	11, 10	
		Vapor NBT 0.000073	12	
		Gas NTP 0.000231	12, 10	

Property Values of Fluorine at Selected Conditions				
Property			Value	Reference
Molecular Weight			37.9968	1
Triple Point Values	Temperature, °F		-363.30	2
	Pressure, psia		0.0321	2
	Density, lb/ft ³	Solid	118.65	3
		Liquid	106.484	3
		Vapor		
Normal Boiling Values	Temperature (T _b), °F		-306.61	2, 3
	Density, lb/ft ³	Liquid	94.08	4
		Vapor	0.3519	a 5
Critical Values	Temperature, °F		-200.47	6
	Pressure, psia		808.5	6
	Density, lb/ft ³		29.44	5
One Gallon Liquid (NBT) Equivalents	Weight, lb		12.58	4
	Volume of gas, ft ³	NBT	35.75	5
		NTP	127.89	b 3
		Equivalent Volumes of Gas per Volume of Liquid (NBT)		NBT 265.78
		NTP 956.83	7, 4	
Heat of Fusion, Btu/lb			TP 5.778	2, 3
Heat of Vaporization, Btu/lb			NBT 73.835	2, 3
Specific Heat Btu/lb-°F	C _p	Solid	TP 0.32132	2
		Liquid	NBT	
		Vapor	NBT	
	C _v	Liquid	NBT	
		Vapor	NBT	
		Gas	NTP	
	C _p	Liquid	NBT 0.36697	2
		Vapor	NBT	
		Gas	NTP	
Specific Heat Ratio	C _p /C _v	Liquid	NBT	
		Vapor	NBT	
		Gas	NTP	
Thermal Conductivity K Btu/hr-ft-°F	Liquid	NBT 0.0915	8	
	Vapor	NBT 0.00416	a 9, 10	
	Gas	NTP 0.01532	9, 10	
Viscosity μ Centipoise	Liquid	NBT 0.243	c 11, 10	
	Vapor	NBT 0.0073	c 12	
	Gas	NTP 0.0231	12, 10	

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