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LIQUEFIED NATURAL GAS RESEARCH

at the

NATIONAL BUREAU OF STANDARDS

PROGRESS REPORT FOR THE PERIOD
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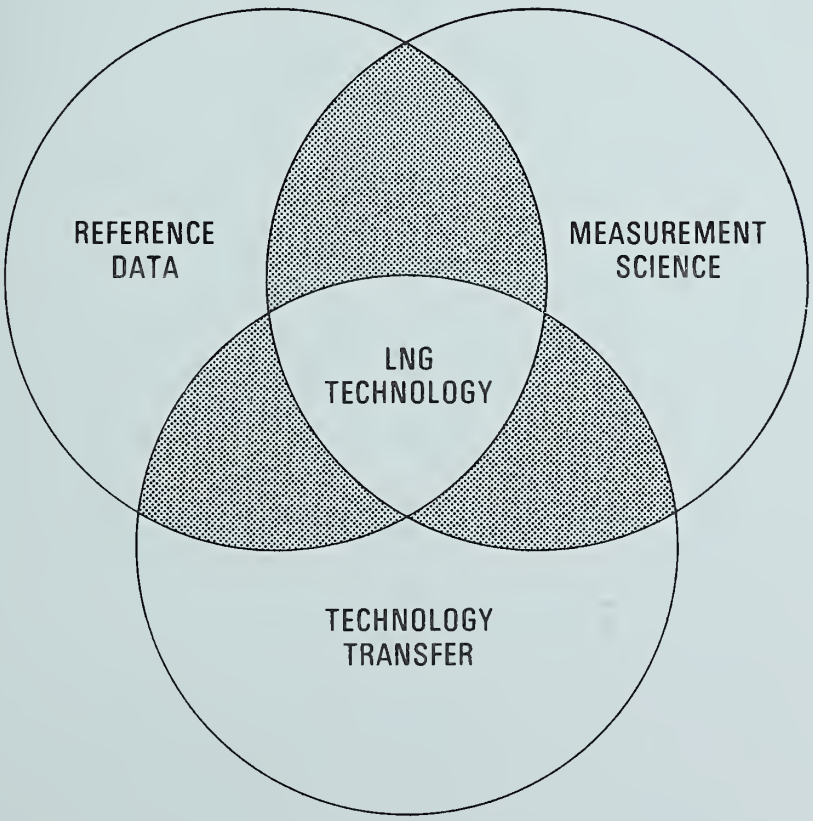

THERMOPHYSICAL PROPERTIES DIVISION, NATIONAL ENGINEERING LABORATORY,
NATIONAL BUREAU OF STANDARDS, BOULDER, COLORADO

NBSIR 79-1617

LIQUEFIED NATURAL GAS RESEARCH
at the
NATIONAL BUREAU OF STANDARDS

Thermophysical Properties Division
National Engineering Laboratory
National Bureau of Standards
Boulder, Colorado 80303

Progress Report for the Period
1 January - 30 June 1979

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ABSTRACT

The objective of this report is to:

1. provide all sponsoring agencies with a semiannual report on the activities of their individual programs;

2. inform all sponsoring agencies on related research being conducted at the NBS-NEL Themophysical Properties Division, NBS-NEL Mechanical Processes Division and NBS-NML Fracture and Deformation Division;

3. provide a uniform reporting procedure which should maintain and improve communication while minimizing the time, effort and paperwork at the cost center level.

The work is supported by NBS and eight other agencies and represents the collective expenditure of $619,000 during the 6-month reporting period. The contents of this report augment quarterly progress meetings for certain of our sponsors and provide a perspective which is missing when the parts are viewed individually. Distribution of this document is limited and intended primarily for the supporting agencies. Data or other information must be considered preliminary, subject to change and unpublished, and therefore not for citation in the open literature.

Key words: Cryogenics; liquefied natural gas; measurement; methane; properties; research.

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1. Title. THERMOPHYSICAL PROPERTIES DATA FOR PURE COMPONENTS AND MIXTURES OF LNG COMPONENTS

Principal Investigators. R. D. Goodwin, H. M. Roder, G. C. Straty, W. M. Haynes, R. D. McCarty, D. E. Diller, and B. A. Younglove

2. Cost Center Numbers. 7360574, 7360548

3. Sponsor Project Identification. Gas Research Institute Grant No. 5010-362-0019 and American Gas Association, Inc., Project BR-50-10. National Aeronautics and Space Administration, Lewis Research Center, Purchase Order C-78014-C.

4. Introduction. Accurate phase equilibrium, equation of state (PVT), and thermodynamic properties data are needed to design and optimize gas separation and liquefaction processes and equipment, and for mass and heat transfer calculations. Accurate data for the pure components and selected mixtures of hydrocarbon systems will permit developing comprehensive accurate predictive calculation methods which take into account the dependence of the thermophysical properties of mixtures on the composition, temperature, and density.

This project will provide comprehensive accurate thermophysical properties data and predictive calculation methods for compressed and liquefied hydrocarbon gases and their mixtures to support the development of LNG technology at NBS and throughout the fuel gas industry. It will also serve as the base for a comprehensive mixtures prediction methodology.

5. Objectives or Goals. The objectives of our work are the determination of comprehensive accurate thermophysical properties data and predictive calculation methods for the major pure components (methane, ethane, propane, butanes, and nitrogen) and selected mixtures of liquefied natural gas and hydrocarbon mixtures at temperatures between 80 K and 320 K and at pressures up to 35 MPa (5000 psi). Our goal is to provide a range and quality of data that will be recognized as definitive or standard for all foreseeable low temperature engineering calculations.

6. Background. Liquefied natural gas is expected to supply an increasing percentage of the United States' future energy requirements. It is likely that massive quantities of liquefied natural gas will be imported during the years 1978 - 1990. Ships and importation terminals are being built for transporting, storing, and vaporizing liquefied natural gas for distribution. Accurate physical and thermodynamic properties data for compressed and liquefied natural gas and hydrocarbon mixtures are needed to support these projects. For example, accurate compressibility and thermodynamic properties data are needed to design and optimize liquefaction and transport processes; accurate data for the heating value, which for liquefied natural gas mixtures depends on the total volume, the density, and the composition, are needed to provide a basis for equitable custody transfer. Accurate mixture data prediction methods are needed for use in automated heat transfer calculations.

Accurate thermodynamic properties data for liquefied gas mixtures must be based on precise compressibility and calorimetric measurements; compressibility data give the dependence of thermodynamic properties on pressure and density (at fixed temperatures); calorimetric data give the dependence of thermodynamic properties on temperature (at fixed pressures and densities). It is impossible, however, to perform enough compressibility and calorimetric measurements directly on multicomponent mixtures to permit accurate interpolation of the data to arbitrary compositions, temperatures and pressures. Instead, thermodynamic properties data for multicomponent mixtures must usually be predicted

(extrapolated) from a limited number of measurements on the pure components and their binary mixtures. This project was initiated to provide the natural gas and aerospace industries with comprehensive accurate data for pure compressed and liquefied methane, the most abundant component in LNG mixtures. We have published National Bureau of Standards Technical Note 653, "Thermophysical Properties of Methane, From 90 to 500 K at Pressures to 700 Bar," by Robert D. Goodwin (April 1974), and National Bureau of Standards Technical Note 684, "Thermophysical Properties of Ethane, From 90 to 600 K at Pressures to 700 Bar," by Robert D. Goodwin, H. M. Roder, and G. C. Straty (August 1976). These reports contain the most comprehensive and accurate tables available for the thermophysical properties of pure gaseous and liquid methane and ethane, and provide an accurate basis for calculating thermophysical properties data for LNG and other hydrocarbon mixtures.

7. Program and Results.

7.1 Propane, PVTx and Dielectric Constant Measurements -- W. M. Haynes

The defective coaxial lead to the capacitor (for dielectric constant measurements) was replaced. However, there have been continuing problems with a low temperature leak in one of the coaxial leads. It is conjectured that this leak was caused by residual acid flux eating a hole in the thin-walled stainless steel sheath of one of the coaxial cables. The leak does not show up at temperatures above 150 K. Therefore, measurements will be performed at temperatures above 150 K before going to the low temperature regime.

After the higher temperature measurements are completed, the apparatus will be disassembled and all coaxial leads will be replaced and encapsulated in high-pressure stainless steel capillaries. This should eliminate the leak problems that have occurred in the past with this type of coaxial lead. This will add additional vapor volume to the cell; however, this presents no problems for pure fluid measurements.

While the apparatus was apart, the design of the capacitor for dielectric constant measurements was changed slightly. The outer cylinder was fabricated into two halves with no slots except for where the cylinder was split. The previous design incorporated the slots as a result of the mixture measurements. The split design allowed the replacement of the coaxial lead without disassembling the guts of the apparatus. It is hoped that this modification reduces the stray capacitance.

For the measurements in the higher temperature region, ethane will be used as a control fluid in aligning the microscope for determination of the position of the buoy. Ethane was chosen for use in this region for several reasons. First, it has sufficient vapor pressure at temperatures above 160 K so that it can be admitted to and removed from the cell easily. Second, precise and accurate data for the density and dielectric constant of saturated liquid ethane are available, most of which were obtained in our laboratory.

At present, some measurements are being made on compressed gaseous methane and nitrogen as further checks on the performance of the capacitor for dielectric constant measurements. After a few measurements on these fluids and tests on liquid ethane are completed, measurements on liquid propane will commence again.

7.2 Calculational Methods -- R. D. McCarty

Work is continuing on the extension and optimization of the corresponding states method to mixtures of methane with nitrogen and other fluids. The transformation of the methane surface to N_2, O_2, Ar, H_2 and C_2H_4 has been the subject of thesis work by a graduate student at the University of Colorado. The work is now complete and is being published as a Masters thesis. We have determined that the transformation function previously used for liquid density near saturation is inadequate for a broader range of pressure and temperature. Efforts in finding a new function are continuing.

7.3 Propane, Specific Heat Data -- R. D. Goodwin

This project is complete and the results published in "Specific Heats of Saturated and Compressed Liquid Propane," by R. D. Goodwin, J. Res. Nat. Bur. Stand. (U.S.) 83, 449-58 (Sep-Oct 1978).

7.4 Normal Butane and Isobutane, Preliminary Thermophysical Properties Data -- R. D. Goodwin

The objective is to prepare provisional tables of thermodynamic properties of normal and isobutane, using available physical properties data. In this work we expect to discover those areas of properties data most in need of further experimental measurements.

In general, there is an abundance of quite inconsistent data from different laboratories, requiring tedious weightings, selections, and eliminations. There are few data for liquid butanes at LNG temperatures.

Satisfactory formulations have been developed for the virial equation, the saturated liquid densities, specific heats for the saturated liquid below the boiling point, and for the ideal gas thermodynamic functions for both fluids.

By thermal loops, in a procedure first developed by W. T. Ziegler, we have derived new "data" (where none existed) from the triple- to the boiling-point for vapor pressures, densities of the saturated vapor, and for heats of vaporization. The vapor-pressure equation has been adjusted to include these new data, and a provisional description has been developed for the heats of vaporization up to the critical point.

Manuscripts for reports on the equation of state and provisional tables of thermodynamic properties for both n-butane and i-butane are complete. The isobutane report is at the printer and the normal butane report is in the final stages of editorial review. Both reports will be published as NBS Interagency reports.

The work on isobutane was done sooner than planned because of a Department of Energy funded experimental project on that fluid which will be used as a geothermal working fluid. The latter work is being done at NBS-Gaithersburg and the two projects will be done on a collaborative basis so that the resulting 'final' tables will serve both projects.

7.5 Pure Nitrogen and Nitrogen-Methane PVTx Property Measurements -- G. C. Straty and D. E. Diller

All planned measurements and data analysis have been completed. A report, PVT Properties of Nitrogen, has been submitted to the Journal of

Chemical Thermodynamics for publication. A second report, PVTx Properties of Nitrogen-Methane Mixtures, is in editorial review and will be submitted to the Journal of Chemical Thermodynamics for publication.

7.6 Sound Velocity of Propane -- B. A. Younglove

Performance of the sound velocity measuring technique has proven satisfactory. No changes in spacer length were required. We have measured sound velocity of the saturated liquid at twenty temperatures between 90 and 300 K. We have also made measurements of sound velocity, temperature and pressure on isotherms at 200, 220, 240, 260, 280 and 300 K. The maximum pressure for these measurements 347 bar (5000 psia).

The measured values of sound velocity have been compared to sound velocity computed from the provisional thermodynamic properties of propane. Deviations as large as 4% have been found.

The next measurements will be on the isotherms at 320 and 340 K followed by a series on the isotherms from 90 to 180 K.

8. Problem Areas. None.

9. Level of Effort. January 1 - June 30, 1979.

Staff-years expended	1.1
Equipment and/or Services Purchased	10.2K$
Approximate expenditures, total	87.4K$ (75.3K$-GRI)

10. Future Plans.

Objectives and Schedule: Quarter	3	4
Optimize existing calculation methods for the thermodynamic properties of methane-nitrogen mixtures and LNG mixtures.	———	
Measure, analyze and report PVT and dielectric constant data for propane.	———	
Publish provisional tables of thermodynamic properties for the butanes and develop an accurate equation of state.	———	
Measure, analyze and report sound velocity data for propane.	———	——→
Publish experimental results for nitrogen and methane-nitrogen mixtures.	———	——→

1. Title. FLUID TRANSPORT PROPERTIES

Principal Investigator. Howard J. M. Hanley

2. Cost Center Number. 7362290, 7362291

3. Sponsor Project Identification. NBS-Office of Standard Reference Data

4. Introduction. Methods for predicting the transport properties of fluid mixtures are unreliable and data are scarce. Prediction methods are needed, however, to supply the necessary design data needed to increase efficiency and reduce costs.

5. Objectives or Goals. The long range or continuing goal of the program is to perform a systematic study of the theories and experimental measurements relating to transport properties, specifically the viscosity and thermal conductivity coefficients, of simple mixtures over a wide range of experimental conditions. The specific objectives of the program include: 1) the systematic correlation of the transport properties of simple binary mixtures and the development of prediction techniques, 2) development of a mixture theory for the dilute gas region and the dense gas and liquid regions, 3) extension of the theory and prediction techniques to multicomponent systems, and 4) suggested quidelines for future areas of experimental work.

6. Background. A continuing program has successfully expanded the state-of-the-art of transport phenomena for pure fluids. Information for pure fluids is required as a prerequisite for mixture studies. The theory of transport phenomena has been developed and applied to produce practical numerical tables of the viscosity, thermal conductivity and diffusion coefficients of simple fluids: Ar, Kr, Xe, N_2, O_2, F_2, He, H_2, CH_4,[3] C_2H_6,[4] C_3H_8.[5]

It has been shown that a successful mixture program can emerge from combining the results for pure fluids with mixture equation of state studies. The equation of state work is being carried out by other investigators in this laboratory.

7. Program and Results. The transport properties of pure ethylene have been computed[6] and a similar correlation of the properties of hydrogen is near completion[7]. We continue to upgrade our mixture prediction procedure reported previously[1]. The thermal diffusivity of mixtures is of interest and work is in progress to prepare a publication of the diffusivity of a methane/ethane mixture at a gas/liquid critical point[2].

Computer simulation of a binary mixture has been carried out, papers are in the press[8].

8. Problem Areas. The lack of suitable experimental mixture transport properties data for comparison purposes is the main problem. Also equation of state (PVT) data for mixtures are needed.

9. Level of Effort. January 1 - June 30, 1979.

Staff-years expended	0.5
Equipment and/or Services Purchased	8.7K$
Approximate expenditures, total	40.5K$

10. Future Plans. The thrust of the effort for the next several months will be to extend the computer simulation studies of mixtures. The studies will yield information on the structure of the mixtures which will, in time, lead to better methods to predict their properties.

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1. Title. PROPERTIES OF CRYOGENIC FLUIDS

Principal Investigators. G. C. Straty, N. A. Olien, B. A. Younglove, H. M. Roder, and D. E. Diller

2. Cost Center Numbers. 7360122, 7360124, 7360125

3. Sponsor Project Identification. NBS

4. Introduction. Accurate thermophysical properties data and predictive calculation methods for cryogenic fluids are needed to support advanced cryogenic technology projects. For example, liquefied natural gas is expected to supply an increasing percentage of the United States' energy requirements through 1990. Liquefaction plants, ships and receiving terminals are being constructed to transport and store natural gas in the liquid state (LNG). Accurate thermophysical properties data for LNG are needed to design low temperature processes and equipment. Accurate data will benefit the energy industries and the consumer by providing for safe and efficient operations and reduced costs. We are now examining the data needs of a number of higher temperature industries such as the synthetic natural gas (SNG) industry. This area of technology as well as the liquefied petroleum gas (LPG) industry are logical extensions of the current LNG work. SNG mixtures can be characterized as much more complex than natural gas, containing unlike (including highly polar) molecules. Interactions between unlike molecules are not well understood and the accurate data necessary to quantitatively understand the interactions are lacking. The needs for accurate predictive methods for SNG are essentially the same as LNG, i.e., to reduce capital and operating costs and improve energy efficiency.

5. Objectives or Goals. The objectives of this project are to provide comprehensive accurate thermodynamic, electromagnetic and transport properties data and calculation methods for technically important compressed and liquefied gases (helium, hydrogen, oxygen, nitrogen, methane, ethane, etc.) at low temperatures. In addition we intend to develop the capability to perform accurate PVT measurements on gaseous mixtures and pure components at high pressures and above room temperature. Precise compressibility, calorimetric and other physical property measurements will be performed to fill gaps and reconcile inconsistencies. Definitive interpolation functions, computer programs and tables will be prepared for engineering calculations. The immediate goals of this work are to obtain accurate sound velocity and thermal diffusivity data for compressed and liquefied gases by using laser light scattering spectroscopy techniques; design, construct and performance test a precision PVT apparatus for the region 250 - 900 K with pressures to 35 MPa; and design, construct and performance test a transient hot-wire thermal conductivity apparatus for the region 70 - 350 K with pressures to 80 MPa.

6. Background. The application of laser light scattering techniques to obtaining thermophysical properties data was initiated to complement and check other measurement methods and to solve measurement problems inherent in more conventional methods. For example, laser light scattering techniques permit measurements of sound velocities for fluids under conditions for which sound absorption is too large to perform ultrasonic measurements; laser light scattering techniques permit measurements of thermal diffusivities under conditions for which convection interferes with measurements of thermal conduction. The feasibility of light scattering experiments to obtain data on binary diffusion coefficients has also been demonstrated.

Light scattering allows thermal diffusivity measurements in the region where density fluctuations are relatively large, but accuracy drops significantly as you pass outside the extended critical region. To complement the scattering method, thermal conductivity measurements can be made with more conventional techniques such as a hot-wire technique. In the latter method a very small platinum wire is surrounded by the fluid and a voltage pulse is applied to the wire. The temperature of the wire is momentarily raised and the resistance increases. A series of very closely spaced resistance measurements would describe the return of the wire to equilibrium. These resistance vs. time measurements can be related to the rate of heat dissipation in the surrounding fluid and thus the thermal conductivity (provided convection heat transfer is prevented).

The development of accurate mathematical models (equation of state) for fluid mixtures requires accurate PVT data for the pure constituents and binary mixtures of key molecular pairs. Experience with LNG has identified the type and accuracy of the data required. In addition to that, work on SNG at high temperatures is a logical follow-on to the low temperature work on LNG. Typical constituents of raw SNG from coal via the Lurgi process are: water - 50.2%; hydrogen - 20.1%; carbon dioxide - 14.7%; carbon monoxide - 9.2%; methane - 4.7%; ethane - 0.5%; hydrogen sulfide and others - 0.6%.

An apparatus has been assembled for laser light scattering spectroscopy measurements on compressed and liquefied gases (76 - 300 K, 35 MPa). The apparatus consists of a high pressure optical cell, a cryostat refrigerated by means of liquid nitrogen, an argon ion laser and low-level light detection equipment.

The light scattered from fluctuations in the fluid can be analyzed with either digital autocorrelation techniques for the examination of the very narrow lines associated with scattering from temperature fluctuations (Rayleigh scattering) or with a scanned Fabry Perot interferometer for the measurement of the Doppler frequency shifts associated with the scattering from propagating density (pressure) fluctuations (Brillouin scattering).

Apparatus for photon-counting and digital autocorrelation has been assembled, interfaced with computer facilities and programmed to enable on-line data accumulation and analysis. Initial problems associated with signal modulations from excessive building vibrations have been solved by levitating the apparatus on an air suspension system. A small, highly stable capacitor has also been designed, constructed and installed inside the scattering cell to permit the dielectric constant of the scattering fluid to be determined, which should allow more accurate fluid densities to be obtained for use in the data analysis. Apparatus tests on well characterized, strongly scattering, test fluids have been made to verify data analysis programs.

Extensive thermal diffusivity data have been obtained for methane. Measurements have been made along the coexistence curve, the critical isochore, and critical isotherm. The measurements extend outside the critical region as well as deep into the critical region. In the deep critical region the effect of temperature gradients and impurities have been investigated. Outside the critical region, these effects do not affect measurements beyond experimental accuracy. The range of the measurements extends from 150 K to 230 K and 3 mol/L to 22 mol/L. The inaccuracy of the measurements is about 5% in the critical region, increasing to 10% or greater further away. A detailed analysis of the data and experimental error has been made and a paper[1] reporting the results has been submitted to the Journal of Chemical Physics.

Some preliminary results on a mixture of 70% methane and 30% ethane were obtained very near the vapor-liquid critical point (plait point). The results are interesting in that the thermal conductivity of the mixture does not exhibit a critical anomaly whereas pure methane does exhibit an anomaly in the thermal conductivity as the critical point is approached. The anomalous behavior of pure fluids and nonanomalous behavior of mixtures is qualitatively and quantitatively in agreement with theoretical predictions (see preceding title 'Fluid Transport Properties'). We hope to be able to perform more definitive measurements on hydrocarbon mixtures in the near future.

7. Program and Results.

7.1 Transient Hot-Wire Apparatus. The assembly of the apparatus is complete and some preliminary measurements on air, nitrogen and helium at low and moderate pressures and near room temperature have been made. Experimental problems have persisted and the new apparatus is still not functioning properly. For the most part computer problems have been solved, but measured values for gas conductivity at low and moderate pressures remain about 8% too high.

7.2 Laser Light Scattering Measurements. This project is inactive at the present time.

7.3 High Temperature PVT Apparatus. Adequate funding for this program was obtained in December 1978; as a result design, procurement and construction will proceed at a level of effort of approximately 1.5 staff-years/year. Nearly all of the required equipment for the new PVT apparatus has been received or is on order. This includes: thermometers, thermometry bridge, desktop computer for experimental control and data acquisition, pressure transducers, high temperature ovens and high temperature pressure cell. The sample cells, thick-wall stainless steel spheres, are nearing completion. During the next few months assembly and checkout can begin. When completed this apparatus and our existing PVT apparatus will give this laboratory the capability of making precision measurements over the temperature range 10 - 1000 K with pressures to 350 bar.

7.4 Analyzing Gaseous Mixtures by Means of Raman Scattering. During the past several months one of our staff members has worked on an experimental program at NBS-Gaithersburg. The objective was to explore the use of Raman spectra in determining accurately the composition of gaseous mixtures such as natural gas. Optical techniques offer the possibility of analyzing gas and vapor compositions in situ. Tests were run using a series of gravimetrically prepared mixtures such as methane-isobutane, methane-nitrogen and an LNG-like 8-component mixture. The staff member has now returned to Boulder, but the preliminary results are very encouraging. We plan a Raman capability in NBS Boulder with the idea of using the principal to make precision determinations of, for example, vapor phase compositions in complex liquid-vapor systems.

8. Problem Areas. Light scattering has proven to be a valuable tool for obtaining thermal diffusivity data on fluids. This is particularly true in a broad temperature and density range around the critical point, where more conventional experimental methods fail or are severely limited. The intensity of the scattered light however decreases drastically as one moves away from the critical region. Data accuracy in this region becomes limited by the statistical nature of the scattering process and the ability to maintain stability and precise experimental parameters over the extended periods of time necessary for data accumulation.

We hope to hire a light scattering expert (experimentalist) in the next few months to help resolve these problems as well as to explore other optical techniques for fluid property studies.

The measurements using the hot-wire apparatus continue to be delayed because of experimental problems.

9. Level of Effort. January 1 - June 30, 1979.

Staff-years expended	2.1
Equipment and/or Services Purchased	27.2K$
Approximate expenditures, total	153.0K$

10. Future Plans.

Objectives and Schedule: Quarter	3	4
Complete performance test transient hot-wire thermal conductivity apparatus.	——→	
Design, construct and performance test high temperature-high pressure PVT apparatus.	————————	

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1. Title. PROPERTIES OF CRYOGENIC FLUID MIXTURES

Principal Investigators. M. J. Hiza, A. J. Kidnay (part-time), and R. C. Miller (part-time)

2. Cost Center Numbers. 7360123, 7362289, 7368574

3. Sponsor Project Identification. NBS, NBS (OSRD), Gas Research Institute

4. Introduction. Accurate thermodynamic properties data and prediction methods for mixtures of cryogenic fluids are needed to design and optimize low temperature processes and equipment. This project provides new experimental measurements on equilibrium properties and compilations of evaluated equilibrium properties data which are suitable for direct technological use or for the evaluation of prediction methods.

5. Objectives or Goals. The overall objectives of this project are to provide critically evaluated data on the phase equilibria and thermodynamic properties of cryogenic fluid mixtures. The program has been divided into the following elements:

a) Preparation of a comprehensive bibliography on experimental measurements of equilibrium properties for mixtures of selected molecular species of principal interest in cryogenic technology.

b) Selection and/or development of methods for correlation, evaluation and prediction of equilibrium properties data.

c) Retrieval and evaluation of experimental data for specific mixture systems selected on the basis of theoretical and/or technological importance.

d) Preparation of guidelines for future research based on the deficiencies noted in (a), (b), and (c).

e) Performing experimental research to alleviate deficiencies and provide a basis for improvement of prediction methods.

6. Background. A physical equilibria of mixtures research project was established in the Thermophysical Properties Division in 1959. The initial effort, based on a bibliographic search and other considerations, was directed toward the acquisition of new experimental data on the solid-vapor and liquid-vapor equilibria and physical adsorption properties for a limited number of binary and ternary mixtures of components with widely separated critical temperatures. Most of the systems studied included one of the light hydrocarbon species -- methane, ethane, or ethylene (ethene) -- with one of the quantum gases -- helium, hydrogen, or neon. The data for these systems led to significant improvements in the predictions of physical adsorption equilibrium and a correlation for the prediction of deviations from the geometric mean rule for combining characteristic energy parameters. In addition, significant new information was obtained for interaction third virial coefficients which was used in a correlation by one of our consultants, J. M. Prausnitz. The approach taken in this work has been as fundamental as possible with the intention of having an impact on a broad range of mixture problems.

Recent efforts have been directed toward problems associated with systems containing components with overlapping liquid temperature ranges, such as nitrogen + methane, methane + ethane, etc.

7. Program and Results. A summary of recent progress is as follows:

a) The paper[1] discussing compilation, evaluation, and correlation of liquid-vapor equilibria data for methane + ethane is in press.

b) A similar paper[2] discussing compilation, evaluation, and correlation of liquid-vapor equilibria data for methane + propane has been sent to the Office of Standard Reference Data for NBS technical review.

c) Based on the work in (b), graphs of K-values and liquid-vapor phase envelopes have been prepared for the second supplement of the LNG Materials and Fluids Handbook.

d) Work on updating and expanding the bibliography[3] on equilibrium properties of fluid mixtures is well underway, though this is no small task. In the original bibliographic effort, we attempted to include references for both saturated and unsaturated hydrocarbons through the C_4's but found that our coverage of unsaturated C_4 hydrocarbons was very incomplete. Therefore, in our current effort we are including all saturated and unsaturated hydrocarbons through C_3, but only the saturated straight and branch C_4's and C_5's (i.e., isobutane, normal butane, isopentane, normal pentane, and neopentane). In addition to the three pentanes, water is also being included which obviously adds a completely new dimension to our effort.

8. Problem Areas. None

9. Level of Effort. January 1 - June 30, 1979

Staff-years expended	0.75
Equipment and/or Services Purchased	3.3K$
Approximate expenditures, total	61.0K$ (12K$-GRI)

10. Future Plans.

Objectives and Schedule: Quarter	3	4
Complete editorial review and prepare final revision for publication of paper reporting compilation, evaluation, and correlation of liquid-vapor equilibria data for methane + propane.	———	→
Continue update and expansion of bibliography on equilibrium properties of fluid mixtures.	———	———

References

1. M. J. Hiza, R. C. Miller, and A. J. Kidnay, A Review, Evaluation, and Correlation of the Phase Equilibria, Heat of Mixing, and Change in Volume on Mixing for Liquid Mixtures of Methane + Ethane. J. Phys. Chem. Ref. Data, 8, No. 3 (1979). In press.

2. R. C. Miller, A. J. Kidnay, and M. J. Hiza, A Review, Evaluation, and Correlation of the Phase Equilibria, Heat of Mixing, and Change in Volume on Mixing for Liquid Mixtures of Methane + Propane, J. Phys. Chem. Ref. Data (in review).

3. M. J. Hiza, A. J. Kidnay, and R. C. Miller, Equilibrium Properties of Fluid Mixtures - A Bibliography of Data on Fluids of Cryogenic Interest, NSRDS Bibliographic Series, IFI/Plenum, New York, 1975.

1. Title. DENSITIES OF LIQUEFIED NATURAL GAS MIXTURES

Principal Investigators. W. M. Haynes, R. D. McCarty and M. J. Hiza

2. Cost Center Number. 7361574

3. Sponsor Project Identification. LNG Density Project Steering Committee, American Gas Association, Inc., Project BR-50-11.

4. Introduction. Accurate density measurements and calculational methods for liquefied natural gas mixtures are needed to provide a basis for custody transfer agreements and for mass, density, and heating value gauging throughout the fuel gas industry.

The basis for the custody transfer of natural gas is its heating value. It is difficult to determine and agree on the heating value of extremely large volumes of natural gas in the liquid state. For example, methods for calculating the heating value of a liquefied natural gas mixture require knowing its density, which in turn depends on its composition, temperature, and pressure. As the compositions of LNG mixtures vary considerably, depending on the sources of the gas and the processing conditions, accurate methods are needed for calculating liquid densities at arbitrary compositions, temperatures and pressures. The accuracy is important because of the extremely large volumes of liquid involved.

5. Objectives or Goals. The objectives of this work are to perform accurate (0.1%) and precise (0.02%) measurements of the densities of saturated liquid methane, ethane, propane, butanes, nitrogen and their mixtures mainly in the temperature range 105 - 140 K, and to test and optimize mathematical models for calculating the densities of LNG mixtures at arbitrary compositions and temperatures.

6. Background. This project is being carried out at NBS because of the realization that equitable custody transfer agreements could be reached more readily if the density measurements and the evaluation and development of calculational methods were performed by independent professionals.

An apparatus incorporating a magnetic suspension technique has been developed for absolute density measurements on liquids and liquid mixtures, particularly at saturation, for temperatures between 90 and 300 K. The estimated imprecision of measurement is less than 0.02% and the estimated inaccuracy is less than 0.15%.

7. Program and Results.

7.1 Measurements. All measurements for this project have been completed. The last series of measurements included the following: (a) two methane-rich binary mixtures of CH_4 and iC_4H_{10}, (b) four methane-rich binary mixtures of CH_4 and nC_4H_{10}, and (c) seventeen methane-rich multicomponent mixtures of LNG components, including five 7- and 8-component mixtures containing up to 0.44% pentanes. The data have been analyzed and comparisons of the experimental densities with calculated values from mathematical models developed for predicting LNG densities have been made.

Papers describing the apparatus, experimental procedures, and the data and its analysis, from the last series of measurements are being prepared for publications.

7.2 Calculational Methods. Final optimization on revision to all of the models is now complete and two manuscripts are in preparation. The final draft of the paper to appear in the Journal of Chemical Thermodynamics is complete and in the review process. The preparation of the NBS document which will contain computer code and detailed instructions on their use is just beginning.

8. Problem Areas. None

9. Level of Effort. January 1 - June 30, 1979

Staff-years expended	0.1
Equipment and/or Services Purchased	5.0K$
Approximate expenditures, total	5.0K$

10. Future Plans. This program is complete apart from the publication of the final results. There are three experimental papers in preparation, an apparatus paper, a paper detailing the performance of the predictive models and a final report encompassing all results of this six year project.

1. Title. PROGRAM FOR REDUCING THE COST OF LNG SHIP HULL CONSTRUCTION -- PHASE II SHIP STEEL IMPROVEMENT PROGRAM

Principal Investigators. H. I. McHenry, M. B. Kasen and R. P. Reed

2. Cost Center Numbers. 5621410, 5621411

3. Sponsor Project Identification. Maritime Administration Misc. P.O. 400-58073.

4. Introduction. Construction of LNG tankers requires the use of fine grain normalized steels for the part of the hull structure that is cooled by the cargo to temperatures in the range of 255 K (0°F) to 228 K (-50°F). Several ABS steels have satisfactory base plate properties but extreme care must be exercised during welding to avoid degradation of the steel adjacent to weld (the heat affected zone) to a level of toughness below U. S. Coast Guard requirements. Significant cost problems are being encountered by U. S. Shipyards due to the resulting inefficient low-heat-input weld procedures that must be employed to meet the fracture requirements in the heat affected zone.

   The feasibility of reducing the cost of LNG ship hull construction was investigated in Phase I of this project, leading to the Phase II program described below.

5. Objective. The objectives of the Phase II program are 1) to have the four major plate producers supply three LNG shipyards with production heats of ABS steels modified to possess improved transverse fracture properties at low temperatures, 2) to have the LNG shipyards evaluate these plates by qualifying optimum welding procedures in accordance with the USCG requirements and 3) to provide a metallurgical evaluation of factors that influence heat affected zone toughness in the improved steels.

6. Background. Early in 1974, the Welding Panel of MarAd's Ship Production Committee recommended that a program be conducted to reduce the cost of ship hull construction. NBS was requested by MarAd to propose such a program to the LNG subcommittee of the Welding Panel at a meeting in Boulder in August. In mid-October, MarAd approved the initial phase of NBS's recommended program, i.e., to survey the problem and the technology available for its solution. On the basis of this survey and as the result of a meeting of the Welding Panel in March, 1975, a coordinated program involving the LNG shipyards, the steel suppliers and NBS was recommended to MarAd and to the Welding Panel. This program was approved and work started in May 1975. Cost-sharing contracts for the evaluation of the improved steels were awarded in 1975 to the three participating shipyards: Avondale shipyards, Inc., Newport News Shipbuilding and Drydock Co. and General Dynamics-Quincy Shipbuilding Division. A similar contract was signed with Lukens Steel Co. to evaluate Cb-treated V-051 steel with and without sulfide shape control. The three participating shipyards and Lukens Steel Co. completed the shipyard evaluation phase of the program in 1977. The ABS steels with sulfide shape control had excellent toughness and the HAZ toughnes was improved, particularly for shielded metal arc (SMA) and gas metal arc (GMA) welds. The most promising results for high-heat-input welds were obtained with the Cb-treated V-051 steel with sulfide shape control.

   Follow-on contracts were awarded to the three participating shipyards to further evaluate the most promising ship steels: ABS grades V-051

(Cb, SSC), V-062 (SSC) and (Cb, SSC). The work on these contracts has been completed. The results indicate that each of the steels retains satisfactory HAZ toughness at -51°C (at higher heat inputs than conventionally used) providing the weld metal toughness exceeds the 27J (20 ft. lb.) minimum.

7. Program and Results. During this reporting period, no additional research was conducted. A presentation of the program results was given at the AWS/MarAd Conference on Welding in Shipbuilding: Productivity and Economics.

8. Problem Areas. The completion of the evaluation of M1-88 wire was delayed due to other shipyard commitments.

9. Level of Effort. January 1 to June 30, 1979.

Staff years expended	0.1
Approximate expenditures, total	$2K

10. Future Plans. An evaluation by Avondale Shipyards Incorporated of submerged arc weldments with Linde M1-88 wire will be completed. Upon completion, the final report on the program will be finished.

1. Title. MATERIALS FOR LNG APPLICATIONS

Principal Investigators. L. L. Sparks

2. Cost Center Number. 7363574

3. Sponsor Project Identification. Gas Research Institute Grant Number 5014-361-0131.

4. Introduction. Safe, economical storage and transportation of LNG depend to a large extend on the materials used in the construction of capital equipment, i.e., ships, storage facilities, transfer terminals, liquefiers, and pipelines. Thermal insulations constitute a critical link in the materials for use in LNG oriented energy efforts because of the cryogenic nature of the liquid.

Expanded plastics (foams) are being used or are being considered for use in facilities such as these. Low-temperature properties data, standardized cryogenic test methods, and standard reference materials are not presently adequate to meet the needs of complicated insulation systems. These materials are anisotropic for most properties so that properties data must be accompanied by specimen characterization in order to be of general use. Standard reference materials, for use in checking experimental systems are completely lacking. With the exception of thermal conductivity, standardized test methods for low-temperature testing of foams are also completely lacking.

5. Objectives or Goals. The broad objectives of this project are twofold: first, provide the users of thermally insulating foams with basic knowledge about the behavior of expanded plastics at cryogenic temperatures and methodology to allow experimental determination of the properties of specific materials; second, serve as a center of insulation material information for GRI and associated users of cryogenic fuels. The first objective will be achieved by determining the low-temperature thermal and mechanical properties of selected, well characterized foams. The methodology of testing at low temperatures and characterizing the test materials will be stressed. The second objective will result as a consequence of this research effort.

6. Background. The Thermophysical Properties Division, previously known as the Cryogenics Division, has been involved in the development and application of cryogenic insulations since being established in 1951.

The Properties of Solids group at Boulder has successfully examined the thermal and mechanical properties of a wide variety of materials: metals, polymers, insulations, and composites. The group, as a whole, has the equipment and expertise to establish and perform cryogenic tests to determine the mechanical and thermal properties of foams. Close working relationships with private companies, universities, and professional associations provide guidance in the areas of material selection, quality control, and standardization of materials and methods.

The groundwork for the current foams research was done while working on the Insulation Section of the LNG Materials Handbook (funded by Maritime Administration).

7. Program and Results. The program designed to meet the need for low-temperature data and standards for expanded plastics includes the following items: assessment of materials, assessment of methods, specimen acquisition, specimen characterization, and testing. A continuing survey of the open literature and contacts with many foam related companies are being used to assess materials and methods.

The cooperation of users, producers, standardization societies, and other government labs, is extremely important to the success of the program. It is being stressed, particularly with the producers, that this program is not intended to do their research for them; out results will be related to identifiable characteristics such as general type of foam, cell size, membrane thickness, manufacturing process, etc. Property dependence on these parameters can be related to new as well as existing products.

Test procedures which have been used are being determined and evaluated. This information along with past experience at NBS-Boulder in cryogenic testing of foams, is being used to establish the initial procedures for this program. Evaluations of these procedures over the duration may lead to extensions and modifications of existing standardized methods to include the cryogenic temperature range.

The characterization of materials will be accomplished by utilizing both inhouse capabilities and that of other government laboratories. Acquisition of equipment which will allow inhouse capability to be developed is being considered; the possibility of interlabatory equipment loans and purchase with general purpose equipment funds are being studied. The procedures, time required, cost, etc. for analysis at other government laboratories are being determined.

The ASTM sanctioned thermal conductivity, , systems are available: a guarded hot plate system, capable of testing from 77 to 1270K has undergone modifications and is now in the checkout stage. The second system is a boil-off calorimeter which allows a number of parameters to be varied while is being determined. Activation of this system is in progress.

Several designs for a general purpose thermal expansion, , system are being considered. Inhomogeneous, anisotropic materials such as foams, aggregates, and fiber-matrix composites require large diameter geometries. Modification of an existing system or design and construction of a new one will be jointly funded by several projects.

8. Problem Areas. None.

9. Level of Effort. January 1 - June 30, 1979

Staff years expended	0.04
Equipment and/or Services purchased	0
Approximate Expenditures, Total	$2,800

10. Future Plans. The assessment phases will continue. Although actual specimen acquisition will not begin until the material assessment phase is completed, the sources and procedures are being developed now. The characterization and testing a particular foam will begin in the next reporting period. The thermal expansion system will be completed and the activation of the boil-off calorimeter system will continue.

1. Title. LNG MATERIALS RESEARCH, CONCRETES

Principal Investigators. L. L. Sparks and M. B. Kasen

2. Cost Center Number. 7360403, 5621511

3. Sponsor Project Identification. Maritime Administration, Miscellaneous Purchasing Order No. 400-89019.

4. Introduction. Concretes are attractive materials for construction of large LNG installations because of lower material cost and economies of fabrications compared to alternative construction methods. They exhibit favorable mechanical and thermal properties for use in LNG applications. Although these materials are presently used to some extent in LNG construction, lack of confidence in their cryogenic behavior has restricted their use to noncritical components or has resulted in overly conservative and expensive designs. This reflects the lack of dependable cryogenic thermal and mechanical properties data and a poor understanding of the effect of field fabrication variables on cryogenic performance.

Applications of special concretes such as lightweight, cellular, fiber reinforced, and polymer will depend on their cryogenic properties, which are unknown at this time. The experimental program to determine these properties will be executed in cooperation with the USCG, Portland Cement Association (PCA), and private corporations such as Global Marine, Concrete Technology, and Preload Technology.

5. Objectives or Goals. The program objectives are to determine the low temperature ($76K \leq T \leq 300K$) properties of concretes which are presently being used in LNG applications and those which may provide design alternatives in the future. The properties which may be determined include: thermal conductivity, Young's modulus, thermal expansion, compressive strength, fatigue strength, splitting strength, permeability to water and LNG, and thermal shock. The dependence of these properties on parameters such as moisture content, air content, water-to-cement ratio, additives, aggregate type and grade, and aging will be determined. The parameter dependencies will be studied in order to understand the fundamental basis for the cause/effect relationships which are observed. Specimen characterization and parameter control are extremly important to this phase of the program. Knowledge gained from the parameter dependencies will be used to assess variabilities found in field-erected structures. This information is needed to establish the quality control necessary to assure fitness-for-service in cryogenic applications. The testing procedures which are used must produce reproducible and accurate data. With the exception of thermal conductivity tests, there are no standardized procedures for testing concretes at cryogenic temperatures. The large variations found in the literature are due, in part, to this deficiency. Whenever possible, ASTM, and ACI procedures, modified to accommodate the cryogenic environment, will be used throughout this program.

6. Background. The initial involvement of the Cryogenics Division in the use of concretes as low temperature structural materials was in 1973. Safety aspects of concrete, cryogenic installations were stressed. Data from the literature were collected and used in these safety evaluations; it became very clear that reliable cryogenic data, basic cause/effect relationships, and standardized cryogenic experimental procedures were inadequate. The situation has not changed significantly, as was determined in a thorough literature evaluation of concrete at cryogenic temperatures (LNG Materials Handbook, second supplement, funded by MarAd).

The Properties of Solids group at NBS-Boulder has successfully examined the thermal and mechanical properties of a wide variety of materials: metals, polymers, insulations, and composites. The group, as a whole, has the equipment and expertise to perform cryogenic tests to determine the mechanical and thermal properties of concretes. Close working relationships with private companies, universities, and professional associations provide guidance in the areas of material selection and quality control of field-erected structures.

7. Program and Results. The specimens to be used for determining the compressive strength have been obtained; the chemical composition of the cement and the composition and size distribution of the aggregates have been determined.

The system needed to measure the thermal contration of these inhomogeneous materials is being designed. Special considerations for this apparatus include the capability to accommodate large diameter specimens (necessary in the case of inhomogeneous materials such as concrete.

Two systems will be utilized to determine the thermal conductivity of concretes. One of these systems is operational while the other is being reactivated.

Specific test procedures are being studied for determination of each of the above properties. It is our goal to develop low-temperature procedures which not only produce accurate results in our tests, but can be adopted by other laboratories.

8. Problem Areas. None.

9. Level of Effort. January 1 - January 30, 1979

Staff years expended	0.04
Equipment and/or services purchased	none
Approximate Expenditures, Total	$4,500

10. Future Plans. Low-temperature compressive strength tests will be completed for the initial concrete mix. Methods of determining the compressive stress-strain relationship will be evaluated.

Design and construction of the system necessary to measure the thermal contraction will continue. Reactivation of the guarded cold plate calorimeter for determining thermal conductivity will continue. The initial thermal conductivity measurements will be made using the smaller guarded hot plate system which is operational at this time.

1. Title. CUSTODY TRANSFER - LNG SHIPS

Principal Investigators. W. C. Haight, R. J. Hocken, B. R. Borchardt, R. G. Hartsock, R. C. Veale, J. D. Siegwarth, J. F. LaBrecque, C. L. Carroll, C. P. Reeve, and F. E. Scire

2. Cost Center Numbers. 7360460, 7361575, 7362575, 7363575, 7311573, 7311577

3. Sponsor Project Identification. LNG Custody Transfer Measurements Supervisory Committee and Maritime Administration Misc. P.O. #400-79005.

4. Introduction. In response to the requests from the U.S. shipbuilding industry, NBS is independently examining the accuracies of LNG tank cargo capacity tables and developing alternative survey techniques.

5. Objectives. The objectives of the program are to develop new techniques for LNG transport tank calibration and to test the accuracy of present calibration techniques as part of an overall study of custody transfer methods aimed at increasing the accuracy of custody transfer measurements.

6. Background. Initial funding by the Maritime Administration (7360460) supported some preliminary tests of calibration of spherical LNG ship tanks. As a result of these measurements, the LNG Custody Transfer Supervisory Committee and the Maritime Administration have funded extension of the work to the membrane tanks and the free standing prismatic tanks now under construction in U.S. shipyards.

7. Program. The NBS measurements of volume of the membrane tanks of all three of the U.S. built El Paso LNG ships have been completed. The NBS determined volume - height relationships for the tanks of the first ship agree with those determined by the primary surveyor to better than 0.2% of total volume. Calibration report have been issued for all tanks of the first ship. Calibration reports on the results of the NBS measurements of the tanks of the remaining two ships are in preparation.

The photogrammetric measurements of the 15 free standing prismatic tanks for the three ships are now completed. A completely independent survey of tank #3 of the third ship has been completed. This tank was also surveyed during the hydrostatic testing to investigate the effect of hydrostatic loading on the tank volume. The dead wood correction, i.e., the removal of volume of aluminum included within the calibrated volume has been examined in detail on this tank. A preliminary report on the volume uncertainty of the tank of the first ship has been supplied to the owner. The tank orientations and bottoms have been surveyed on the first two ships.

8. Problem Areas. None

9. Level of Effort. January 1 - June 30, 1979

Staff years expended	1.5
Equipment and/or services purchased	37 K$
Approximate expenditures, total	91 K$

10. Future Plans. Measurements of the orientation and bottom surveys of the tanks of the El Paso Columbia remain to be done. Tank calibration report on the remaining two membrane tank ships and the preliminary uncertainty report on the final two ships with free standing prismatic tanks will be completed. The results of the additional surveys on the free standing tanks will be documented and a final report issued on the tank calibration and the photogrammetric methods.

1. Title. HEATING VALUE OF FLOWING LNG

Principal Investigators. J. A. Brennan

2. Cost Center Number. 7362570

3. Sponsor Project Identification. Pipeline Research Committee (American Gas Association) PR-50-48.

4. Introduction. This project will test instrumentation for making heat value measurements on flowing LNG in actual applications. Information from projects currently underway by Siegwarth (cost center 7367574) on densimeters, by Haynes and Hiza (cost center 7361574) on mixture densities and by Richards (cost center 7363570) on LNG sampling will be utilized where appropriate to provide state of the art information.

5. Objectives. The objective of this project is to measure total heating value of LNG flowing in a pipeline by the integration of individual measurements of flow, density and specific heating value. Flow measurement requires determination of flowmeter performance in line sizes larger than are presently available in operating calibration facilities. Therefore, a secondary objective is to establish appropriate flowmeter scaling laws.

6. Background. The LNG flow facility at NBS was used to evaluate the response and integration of the individual elements of the heating value measurement. Different compositions of LNG were tested to provide a range of densities and temperatures sufficient to determine any dependencies. Sampling work was combined with the sampling project (cost center 7363570) to better define the important criteria of this phase of the measurement problem.

   Flowmeter scaling work utilizes the cryogenic and water flow facilities at NBS as well as private LNG peak shaving and import facilities.

7. Program and Results. On three different occasions LNG flowmeter tests were run at Southern Energy Company's import terminal. These tests covered a range of flow rates from 88.5 m^3/s (270 x 10^6 SCFD) to 140.9 m^3/s (430 x 10^6 SCFD). During a portion of each test, there were intervals during which the signal from the flowmeter measuring the LNG flow was of very poor quality and resulted in poor agreement between the liquid and gas flow meausrements. Attempts to isolate this problem by changing electronic components was not successful.

   When the flowmeter signal was of good quality, the agreement between the liquid and gas flow measurements was very good. During all of these tests, when the flowmeters were operating correctly, the agreement between the two measurements was within the uncertainty of the gas flow measurement alone.

   Densimeter tests were also run during the flowmeter tests. It was necessary to change some of the electronic components before the densimeter would operate consistently. These changes were completed during the second test and appeared to correct the problem. Data from the densimeter is still being analyzed and will be included in the next report.

   At the present time, no additional testing is planned.

8. Problem Areas. None

9. Level of Effort. January 1 - June 30, 1979

Staff years expended	0.2
Equipment and/or services purchased	none
Approximate Expenditures, Total	$9031.00

1. Title. LNG DENSITY REFERENCE SYSTEM

Principal Investigators. J. D. Siegwarth and J. F. LaBrecque

2. Cost Center Number. 7367574

3. Sponsor Project Identification. American Gas Association, Inc., Project BR-50-10; National Bureau of Standards, Gas Research Institute.

4. Introduction. A density reference system has been developed to evaluate the ability of commercially available instruments to measure densities of LNG directly. Density is an essential measurement in determining the total energy content of natural gas reservoirs.

5. Objectives. The object of this research is to develop and supply adequate calibration methods and calibration standards to densimeter manufacturers and users for providing traceability of accuracy to field density measurement systems.

6. Background. The density reference system project was initiated in 1973. Since that time the reference system has been designed, constructed, and is now in operation, evaluating commercial density metering systems. Reports describing the density reference system and the results of the tests of four commercial densimeters have been published. These reports are:

Siegwarth, J. D., Younglove, B. A., and LaBrecque, J. F., Cryogenic fluids density reference system: provisional accuracy statement, National Bureau of Standards (U.S.) Technical Note 698, 24 pages (1977), and

Siegwarth, J. D., Younglove, B. A., and LaBrecque, J. F., An evaluation of commercial densimeters for use in LNG, National Bureau of Standards. (U.S.) Technical Note 697, 43 pages (1977).

The work has also been presented in the following papers:

Siegwarth, J. D., Younglove, B. A., and LaBrecque, J. F., Test of densimeters for use in custody transfer of LNG, Proc 53rd International School of Hydrocarbon Measurement, Normon, Oklahoma (1978).

Parrish, W. R., Brennan, J. A., and Siegwarth, J. D., LNG custody transfer research at National Bureau of Standards, American Gas Association Operating Section Proc. T243 (1978).

7. The rebuild of the DRS has been completed and test work on three displacement type densimeters and a vibrating cylinder densimeter is underway. The sample holder can now be cooled and filled in about two hours, the temperature differences between top and bottom thermometers can be held below .020 K during the measurements with little difficulty without using the stirrer running at low speed. In fact, it is possible to obtain this degree of isothermal condition at various temperatures without using the stirrer at any time.

Three other vibrating cylinder densimeters are on hand to be tested or calibrated.

A prototype portable standard using a more compact electronic balance and a silicon single crystal has been designed and is under construction.

A calibration method for displacement type densimeters, using pure methane and a methane-propane-nitrogen mixture, is being tested. Preliminary results indicate the method will adequately calibrate densimeters.

The results of the vibrating cylinder densimeter tests to date show the calibration of the densimeter is particularly sensitive to sample composition.

8. Problem Areas. The DRS densimeter readings are shifted about 0.05% relative to the density readings prior to the rebuild of the DRS according to the density determined from the average sample temperature. This systematic difference probably arises from a peculiarity in the electronic balance. This problem will be examined further when the DRS densimeter using the compact balance is operational.

9. Level of Effort. January 1 - June 30, 1979

Staff Years Expended	0.4
Equipment and/or Services purchased	$ 8,000
Approximate Expenditures	$30,000

10. Future Plans. The DRS densimeter using the compact electronic balance will be completed and tested as soon as evaluation of the densimeters presently under test are completed. Besides calibrating two more vibrating cylinder densimeters for transfer standards, a vibrating plate densimeter will also be calibrated as a transfer standard. Arrangements are underway to do additional tests of a capacitance type densimeter.

1. Title. LNG SAMPLING MEASUREMENT STUDY

Principal Investigators. R. J. Richards, J. F. LaBrecque, and J. A. Brennan

2. Cost Center Numbers. 7363570

3. Sponsor Project Identification. Pipeline Research Committee (American Gas Association) PR-50-94.

4. Introduction. Composition is used to determine both the heating value and the quantity (through density) of LNG shipments. Thus, any error in composition doubles when calculating the total heating value and dollar value of a LNG tanker cargo. Compositions are determined by sampling LNG, on either a batch or continuous basis, and analyzing the vaporized mixture. Although several sampling techniques exist, none have received a widespread acceptance in the LNG industry. Also, a standard technique has not been established for analyzing the vaporized sample.

5. Objectives. The objective of this project is to expand the work previously completed on the NBS LNG sampling system. This new work will investigate new vaporizer designs and test variations in accumulator size requirements. The goal is to obtain information necessary for design purposes.

6. Background. This work was performed because there is a need to determine the best means for obtaining the composition of LNG shipments. Current LNG buying contracts include specifications on when and how many liquid samples are to be taken but omit the sampling technique to be used. The evaluation of sampling techniques by NBS is expected to lead to the acceptance of the most accurate composition determination method by all parties involved in LNG custody transfer.

7. Program and Results. As stated in the last report, the final testing on this project was to be on accumulators of various sizes. This was to be done in an effort to reduce the size of the accumulator so that the system time constant could be as short as possible. These tests were completed using six different size accumulators ranging from 0.1 to 4.8 liters. No detectable difference in performance was observed with accumulators with volumes of 1.7 liters or larger. There did appear to be a significant decrease in precision while using accumulators with volume of 0.1 and 0.2 liters, however.

This work is now complete and a final report has been drafted.

8. Problem Areas. None.

9. Level of Effort. January 1 - June 30, 1979

Staff years expended	0.4
Equipment and/or services purchased	none
Approximate Expenditures, Total	$17,250.00

1. Title. SURVEY OF CURRENT LITERATURE ON LNG AND METHANE

Principal Investigator. Neil A. Olien

2. Cost Center Number. 7369574

3. Sponsor Project Identification. Gas Research Institute Grant No. 5010-362-0019 and American Gas Association, Inc., Project BR-50-10.

4. Introduction. It is important that all NBS personnel working in LNG, as well as the AGA and others, keep up with what is going on throughout the world in the LNG field. This project is designed to provide the Current Awareness and other information services to allow workers to keep abreast of new research and other developments.

5. Objectives or Goals. We will publish and distribute each April, July, October, and January a listing of all significant papers, reports, and patents relating to methane and LNG properties and technology. The references will be listed under convenient subject headings. The Quarterly will be distributed to all interested AGA member companies and be made available to the general public on a subscription basis. In addition, LNG-related information will be entered into the Cryogenic Data Center's Information System for quick retrieval. A systematic review of the current publication scene is maintained for any new periodicals to be reviewed cover-to-cover. Finally we will update and make available comprehensive bibliographies on the properties and technology of LNG. There are four bibliographies involved: methane properties, methane mixtures properties, processes and equipment involving methane and LNG, and patents relating to methane and LNG technology. These four will be updated annually.

6. Background. In 1969 we made a thorough review of the world's publications to determine which periodicals and abstracting services should be scanned cover-to-cover to adequately encompass the LNG field. The result is that we now scan over 330 primary publications and nearly 25 secondary publications. Of these, approximately one-third are directly related to LNG. In addition, we have increased our coverage of the energy field to include SNG (coal gas, hydrogen, etc.). Much of this information is also pertinent to LNG and as such is listed in our LNG-related publications. Our Current Awareness Service has been published weekly since 1964 (beginning in 1975 the publication became biweekly) and the Liquefied Natural Gas Survey has been published quarterly since 1970.

7. Program and Results. Four issues of the LNG Quarterly are prepared each year and distributed. There are now 137 subscriptions going to AGA Member Companies and 159 to other subscribers.

The four comprehensive bibliographies mentioned in section 5 have been reviewed and shortened, and more selective bibliographies have resulted. The latest versions were completed as of January 21, 1977.

B-1525 THE THERMOPHYSICAL PROPERTIES OF METHANE AND DEUTERO-METHANE IN THE SOLID, LIQUID AND GASEOUS PHASES - A SELECTED BIBLIOGRAPHY. Indexed by property, phase and author, 100 pages (Jan 1977). ($10.00).

B-1526 THE THERMOPHYSICAL PROPERTIES OF METHANE MIXTURES - A SELECTED BIBLIOGRAPHY. Indexed by property, system and author, 166 pages (Jan 1977). ($15.00).

B-1524 PROCESSES AND EQUIPMENT INVOLVING LIQUEFIED NATURAL GAS AND METHANE - A SELECTED BIBLIOGRAPHY. Indexed by subject and author, 285 pages (Jan 1977). ($25.00).

B-1527 PATENTS RELATING TO METHANE AND LNG TECHNOLOGY - A SELECTED BIBLIOGRAPHY. Indexed by author, 150 pages (Jan 1977). ($15.00).

Over the past seven years we have distributed over 500 copies of these and the comprehensive bibliographies.

The size of these has grown to the extent that their usefulness is limited, therefore it was decided that they would not be updated and a series of more specialized bibliographies would be made available. The following list gives those currently available. Additional topics will be added as necessary.

B-1528 LIQUEFIED NATURAL GAS STORAGE INCLUDING INSULATION SYSTEMS. 1181 references, indexed by author and subject, 210 pages ($20.00).

B-1529 LIQUEFIED NATURAL GAS PEAKSHAVING AND SATELLITE OPERATIONS. 221 references, indexed by author, 36 pages ($10.00).

B-1530 LIQUEFIED NATURAL GAS STRATIFICATION AND ROLLOVER. 38 references, indexed by author and subject, 10 pages ($5.00).

B-1531 LIQUEFIED NATURAL GAS SAFETY INCLUDING SPILLS. 368 references, indexed by author and subject, 72 pages ($10.00).

B-1532 LIQUEFIED NATURAL GAS PIPELINES AND TRANSFER LINES. 226 references, indexed by author, 44 pages ($10.00).

B-1533 LIQUEFIED NATURAL GAS SHIPS, BARGES AND OVERWATER TRANSPORTATION. 805 references, indexed by author, 138 pages ($15.00).

B-1534 LIQUEFIED NATURAL GAS VAPORIZORS INCLUDING COLD UTILIZATION. 330 references, indexed by author and subject, 58 pages ($10.00).

B-1535 LIQUEFIED NATURAL GAS HEAT TRANSFER. 155 references, indexed by author, 34 pages ($10.00).

B-1536 LIQUEFIED NATURAL GAS LIQUEFACTION AND REFRIGERATION. 551 references, indexed by author, 94 pages ($15.00).

B-1537 LIQUEFIED NATURAL GAS ECONOMIC FACTORS. 470 references, indexed by author, 84 pages ($15.00).

B-1538 LIQUEFIED NATURAL GAS OVERLAND TRANSPORTATION. 76 references, indexed by author, 15 pages ($5.00).

B-1539 LIQUEFIED NATURAL GAS PATENTS. 747 references, indexed by author and subject, 119 pages ($15.00).

B-1540 LIQUEFIED NATURAL GAS INSTRUMENTATION. 85 references, indexed by author and subject, 23 pages ($5.00).

The above thirteen bibliographies are in the process of being updated and will be available in August.

Problem Areas. None.

9. Level of Effort. July 1 - December 31, 1978.

Staff-years expended	0.25
Equipment and/or Services Purchased	2.2K$
Approximate expenditures, total	7.5K$

10. Future Plans. Issue 78-4 was delivered to the printer the second week of January and should be distributed before the end of January.

1. Title. LIQUEFIED NATURAL GAS TECHNOLOGY TRANSFER

Principal Investigators. D. E. Diller, H. M. Ledbetter, L. L. Sparks, and N. A. Olien

2. Cost Center Numbers. 7360403, 7361403, 7364574, 7368574, 7360127, 7360461, 7360594

3. Sponsor Project Identification. Maritime Administration, Miscellaneous Purchase Order No. 400-79005; American Gas Association, Inc. Project BR-50-10; Gas Research Institute; NBS Office of Standard Reference Data.

4. Introduction. The liquefied natural gas program at the Thermophysical Properties Division of NBS Boulder represents an investment by industry and government agencies of over $7 million over the past six years. This investment was designed to develop reference quality properties data for both fluids and materials and instrumentation and measurement technology for the use of the LNG and related industries. Information developed under this program must be transmitted to the ultimate user in a timely and useful format. The classical publication methods of NBS most certainly provide the scientist and research engineer information in a form most useful to the academic or near academic community. However, as a result of extensive assessments of user requirements, it was found that an additional effective mode for technology transfer would be an LNG Materials and Fluids User's Manual. A complete outline and planned table of contents have appeared in previous semiannual reports. The Maritime Administration of the Department of Commerce and the American Bureau of Shipping agreeded to sponsor the first year's efforts on the materials section, and the American Gas Association, Inc. and the NBS Office of Standard Reference Data agreeded to sponsor the section on fluids and fluid mixtures. The project was begun on April 1, 1976.

5. Objectives or Goals. The Liquefied Natural Gas Materials and Fluids User's Manual will provide a method of quick dissemination of property data and related information for the effective generation, utilization and transportation of LNG. The object is to improve technology transfer from the current NBS Thermophysical Properties Division LNG physical measurements program to the users, including federal agencies, the states and industry. For the purpose of this data book, liquefied natural gas is defined as a cryogenic mixture (at less than approximately 150 K) of hydrocarbons, predominantly methane, with less than a total of 20% of the minor components ethane, propane, iso and normal butane, and nitrogen as an inert contaminant. LNG materials will be those associated with the liquefaction, transport and storage of liquefied natural gas.

6. Background. The User's Manual is only one of a number of information dissemination methods used to provide workers in the liquefied natural gas (LNG) industry with properties data of known quality in a format consistent with the requirements of the intended user. In the case of the LNG User's Manual the intended audience is the field engineer, plant manager, ship designer or process engineer interested in a ready reference of assessed quality for data to be used in conceptual design, process monitoring, process analysis, and intercomparisons where precision and accuracy are secondary to specific problem solutions. The hierarchy of accuracy and precision will be defined and traceable through references to scientific and engineering literature.

Data are classified into three groups by the NBS Thermophysical Properties Division.

Group 1. Data which have been generated experimentally by NBS, or have been assessed, evaluated or experimentally verified by NBS.

Group 2. Data which have been assessed and evaluated by NBS.

Group 3. Data available in the scientific engineering literature through the NBS Cryogenic Data Center or elsewhere. No NBS evaluation or assessment has been made at this date.

In general, most data included in the LNG User's Manual will be from groups 1 and 2. Few new assessments or correlations are anticipated or required for this work.

Data will be presented primarily in graphical form. Tables and analytical expressions will be used only where absolutely necessary. Graphs and charts will be in loose-leaf form for ease of updating and additions. This form will also allow immediate implementation for data already available under the NBS LNG program and will provide a convenient format for the output of data from existing projects. The User's Manual will not be a substitute for traditional publications in the scientific literature where measurement science, technique, precision and accuracy are paramount, but will provide the data and references for the necessary assessment by the user.

The publication of both graphical and tabular data will be in a dual system of physical units. These units will be the traditional LNG industry British System of BTU, pound, degree Fahrenheit and the SI system of joule, kilogram and kelvin. It is the intent to give equal weight to each system of units.

7. Program and Results. The first edition of the User's Manual became available for distribution in September 1977. A complete description and ordering information are included as part of this report. Over 750 copies have now been distributed to sponsors and purchasers. New orders are currently coming in at the rate of about ten per week. The first supplement to the User's Manual is printed and 350 copies have been distributed

On January 24, 1979 the LNG Materials and Fluids User's Manual received the Award of Distinction (first place) of the Society for Technical Communications. This award was in the category "Industrial Handbooks and Manuals" and we believe recognized the efforts and contributions of the sponsors and individual contributors.

Due to an initial uncertainty about the demand for the User's Manual only 750 of the cover and dividers were ordered, however 1000 copies of all graphs and text were printed. Our original stock of 750 was exhausted in April, therefore 250 additional covers and divider sets were ordered and are on hand for filling orders.

The second supplement is nearing completion. After some difficulty with extremely high bids, a reliable printer was engaged. The second supplement will contain approximately 20-25 graphs on composites, 6-10 on concrete, 15 updated graphs on structural materials, 15 graphs and 3 wall charts on propane properties and three graphs on mixture properties.

8. Problem Areas. None.

9. Level of Effort. January 1 - June 30, 1979

Staff-years expended	1.3
Equipment and/or Services Purchased	22.3K$
Approximate expenditures, total	95.0K$

10. Future Plans. Work on the second supplement is in progress. Two groups of originals will go to the printer in July and the last group in mid-September. We hope to have the printed copies back in late November for distribution.

1. Title. OIML JOINT SECRETARIAT ON LNG MEASUREMENTS

Principal Investigators. Douglas B. Mann and James A. Brennan, NBS and T. L. Hillburn, Phillips Petroleum Company.

2. Cost Center Number. 7360290

3. Sponsor Project Identification. American Gas Association, Inc., NBS-Office of International Standards; and NBS.

4. Introduction. The liquefied natural gas program of the National Bureau of Standards Cryogenics Division has, over the past seven years, provided the gas industry and interested Government agencies with properties data on materials and fluids, instrumentation, and measurement assistance in supplementary fossil energy supply. Support of this program by the American Gas Association, Inc., and Federal Government agencies such as the Maritime Administration (MarAd), NASA, GSA, Federal Power Commission and the NBS-Office of Standard Reference Data has provided a basis for the national acceptance of the results of the NBS LNG program. Through the U.S. membership in the International Organization of Legal Metrology there exists, at the present time, an opportunity to extend, internationally, the utility of data and measurement practice developed under our joint Government/industry program. We have been requested (by OIML membership) to establish a LNG Measurement Secretariat within OIML which, if implemented, would provide a significant international forum for the results of our joint work. It is believed that a joint Secretariat with the LNG industry would provide the most effective means of accomplishing these objectives.

5. Objectives or Goals. Our objective is to accomplish the following goals.

a) To establish U.S. (NBS) thermophysical properties data for LNG as the standard data in international usage.

b) To establish U.S. (NBS) materials property data used in fabrication and construction of LNG facilities (liquefiers, storage, transport) as the standard data in international usage.

c) To establish U.S. (NBS) approved measurement technology and instrumentation as related to LNG (pressure, temperature, density, liquid level, flow) as the standard in international LNG trade. The precedent has been established with the successful completion of the joint NBS-CGA cryogenic flow measurement program which has resulted in the adoption of a cryogenic flow measurement code by the National Conference on Weights and Measures. We wish to extend this code on an international basis.

d) To establish and maintain the leadership of U.S. science, engineering, and industry in the research, technology, manufacture and marketing of instruments and measurement systems for liquefied natural gas.

6. Background. OIML was founded in 1955 to promote intergovernmental cooperation in the field of legal metrology which relates to the compatibility of standards of measurement and the legislation and government regulations which may affect such standards of measurement. OIML recommends uniform international requirements for scientific and measurement instruments used in industry and commerce and works out model laws and regulations for consideration by member nations; and, in

addition, serves as a center of documentation and information exchange in legal metrology. At present, 43 nations are members of this intergovernmental organization.

The United States joined OIML in 1972 (the Senate by resolution of August 11, 1972, gave its advice and consent to the accession of the U.S. to the convention establishing OIML). The responsibility for managing U.S. participation in OIML was assigned to the Department of Commerce and has since been delegated by the Department to the National Bureau of Standards (NBS). Under the general guidance of the Department of State and the Secretary of Commerce, NBS is directly responsible for formulating and implementing U.S. policy towards OIML. U.S. participation in the organization is deemed important for two reasons: First, to protect and enhance some $1 billion worth of scientific and measurement instruments exported each year by U.S. firms and to ensure equity in the trade of commodities measured by these instruments; and second, to maintain the U.S. as the world leader in the field of metrology.

In the spring of 1975 at a meeting in Paris of the International Committee of Legal Metrology, the French and U.S. representatives discussed the possibility of creating a new Reporting Secretariat No. 15 on "Liquefied Natural Gas (LNG) Measurement." The U.S. representative, W. E. Andrus, Jr. of NBS, agreed to explore the possibility with U.S. industry and interested government agencies. These discussions resulted in a decision to propose a joint Secretariat with the American Gas Association and NBS-Cryogenics Division in order to best accomplish the tasks. These conclusions were reached during several meetings extending through the latter part of 1975 and early 1976. During the summer of 1977, representatives of NBS met with PTB (West Germany) and SIM (France) to discuss the proposed scope and to explore expansion of the effort to include cryogenic fluids in general. Results of those discussions indicate some resistance to including different physical measurements (flow, density, etc.) under a single recommendation for a specific group of fluids.

The proposed plan and scope were presented at the meeting of The Advisory Committee for International Legal Metrology held at NBS-Boulder in September. The committee encouraged NBS to proceed with the present scope and to continue to explore the possible inclusion of other cryogenic fluids.

A revised work plan for cryogenic fluids was generated by the technical associates Douglas B. Mann of NBS and T. L. Hillburn of the Phillips Petroleum Co. representing A.G.A. and API. The scope of the work plan included instrumentation and procedures for the custody transfer measurements of pressure, temperature, density, liquid level, flow and calorific value of liquefied atmospheric and natural (hydrocarbon) gases having pure fluid or mixture normal boiling points of less than 150 K. Recommendations will be limited to establishing total mass and, where applicable, total heating value. Fluids and fluid mixtures considered will be limited to commercially important liquefied atmospheric gases, atmospheric gases, oxygen, nitrogen and argon and the primary components of liquefied natural gas, methane, ethane, propane, iso- and normal butane and pentane.

Flow measurements of pure cyrogenic fluids will be the first of the recommendations generated under the proposed work plan. This will be accomplished by combining the existing U.S. and European codes for flow measurements. A working group has been formed of interested parties and

the first draft of this recommendation should be ready for review by January of 1980.

7. Program and Results. The two documents to be combined are:

"Cryogenic Liquid Measuring Devices," NBS Handbook 44, 4th Ed. (with supplements), National Bureau of Standards, Washington D.C. (1976)

"Code of Practice - Metering of Cryogenic Liquids," IGC Industrial Gases Committee, 32, Boulevard de la Chapelle, 75880 Paris Cedex 18, France (October 1976)

Funding delays have shifted the program to the latter half of 1979. Initial outlines of the two codes have been completed, but detailed synthesis have not begun.

8. Problem Areas. Funding delay has extended schedule, but does not cause additional problems.

9. Level of Effort.

Staff-years expended	0.06
Equipment and/or Services purchased	0.00
Approximate Expenditures	$5,000

0. Future Plans. A first draft recommendation will be prepared by NBS as a simple combination of the two existing codes. This draft will be distributed to the four members of the working group for review. The revised first draft will then be submitted to the NBS-OIML representatives for OIML distribution.

1. Title. LNG SAFETY

Principal Investigators. Thomas R. Strobridge

2. Cost Center Number. 7360450

3. Sponsor Project Identification. Lawrence Livermore Laboratory (LLL) order No. EY-78-X-03-029 (3/3/78)

4. Introduction. Small experimental spills of LNG have been conducted on both land and water. A number of mathematical models have been devised describing pool size, spread rate, evaporation rate and plume dispersion encorporating various descriptions of the surface on which the spill occurs and various atmospheric conditions. Lawrence Livermore Laboratory has a program funded by DOE to conduct large spill tests on both land and water complimented by the appropriate instrumentation to refine the measured characteristics of spills and the resulting plume, thus providing improved and expanded data for further modeling efforts. Recently, the LLL program was expanded to possibly include LPG, propane, butane anhydrous ammonia and hydrogen.

5. Objectives and Goals. The objective of this project is to provide consultation to LLL on an as-needed basis regarding safety, equipment and engineering for low temperature applications.

6. Background. NBS has been active in safety programs for the combustible cryogenic fluids for over 30 years. Most recently the major effort has been on LNG safety.

7. Program and Results. LNG storage vessels will be needed at the spill test site. LLL has possession of two 28,000 gallon liquid oxygen dewars that have been used for liquid nitrogen and are currently mothballed. We have recommended the procedures and requirements for inspecting, testing, converting and recertifying the dewars for LNG service.

An extensive literature search of ten data bases was conducted in anticipation of experiments on LPG, propane, butane and anhydrous ammonia spills. Those references suggested for the LLL spill library were selected to give:

1. Thermodynamic and transport properties necessary for process design, instrument selection and data analysis,

2. Materials compatibility for component selection,

3. References to applicable codes and standards,

4. Fundamental studies of combustion, plume dispersion and spills, and,

5. General safety and accident reports.

There are no plans at the present time to combine the results of the various literature searches in a form for public release.

8. Problem Areas. None

9. Level of Effort. January 1 - June 30, 1979

Scaff years expended	0.1
Equipment and/or Services Purchased	-0-
Approximate Expenditures, Total	$7,500.00

10. Future Plans. Provide consulting services as needed.

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See page ii

13. Type of Report & Period Covered: 1 Jan 1979-30 June 1979

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SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

The objective of this report is to:

1. provide all sponsoring agencies with a semiannual report on the activities of their individual programs;
2. inform all sponsoring agencies on related research being conducted at the NBS-NEL Thermophysical Properties Division, NBS-NEL Mechanical Processes Division and NBS-NML Fracture and Deformation Division;
3. provide a uniform reporting procedure which should maintain and improve communication while minimizing the time, effort and paperwork at the cost center level.

The work is supported by NBS and seven other agencies and represents the collective expenditure of $619,000 during the 6-month reporting period. The contents of this report augment quarterly progress meetings for certain of our sponsors and provide a perspective which is missing when the parts are viewed individually. Distribution of this document is limited and intended primarily for the supporting agencies. Data or other information must be considered preliminary, subject to change and unpublished, and therefore not for citation in the open literature.

KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

Cryogenics, liquefied natural gas; measurement; methane; properties; research

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