Thermophysical Properties DIVISION

Early LNG Program and LN_2 Cryogenic Flow Measurement Facility at NIST

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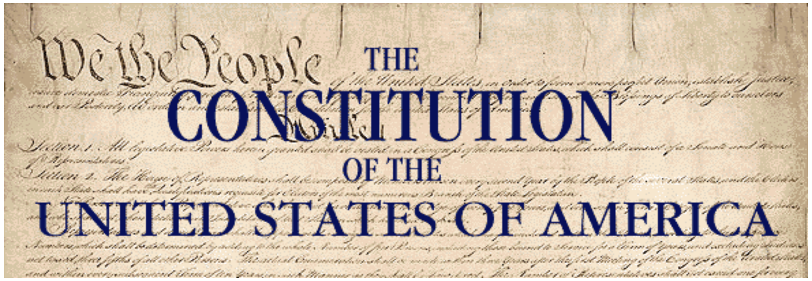
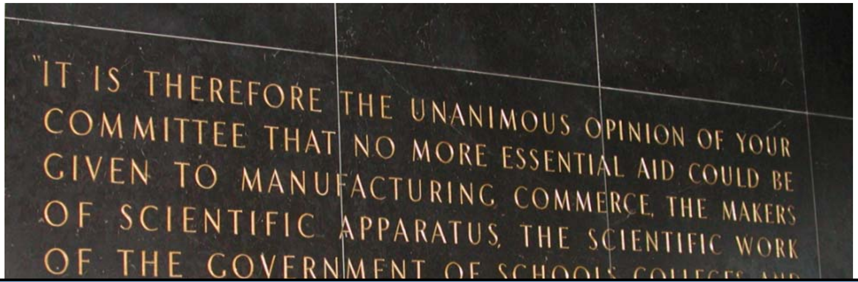
National Institute of Standards and Technology (NIST)

NIST is the National Metrology Institute (NMI) for the U.S., and signatory to the CIPM MRA

- Non-regulatory agency within U.S. Department of Commerce
- Founded in 1901 as National Bureau of Standards



Article I, Section 8: The Congress shall have the power to …*coin money, regulate the value thereof, and of foreign coin, and fix the standard of weights and measures*



Mission …

to promote U.S. innovation and industrial competitiveness by advancing

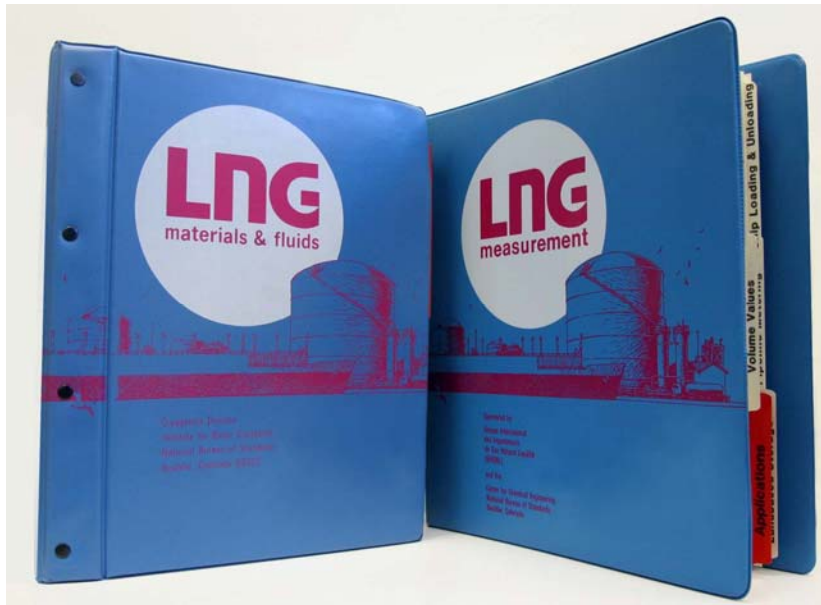
measurement science, standards, and technology

in ways that enhance economic security and improve our quality of life

LNG at NIST (NBS):1970 to 1985

Sponsors and Collaborators

- •Groupe International des Importateurs de Gaz Naturel Liquéfié
- •American Gas Association
- •Gas Research Institute
- •DOC Maritime Administration
- •American Bureau of Shipping
- • …



- •Western LNG Terminal Assoc.
- •Tokyo Gas Co.
- •Columbia LNG Corp.
- •El Paso LNG Co.
- •Ruhrgas LNG
- • …

LNG Materials and Fluids

- Materials
  - Structural Materials: Aluminum Alloys; Invar; Nickel Steels, …
  - Thermal Insulators: Polystyrene; Polyvinyl Chloride; Balsa, …
  - Concrete: Elastic (Young's Modulus); Thermal; Mechanical
  - Nonmetallic Laminates: Cotton fabric/phenolic; glass mat/epoxy
- Fluids
  - Pure Fluids: methane, ethane, … (charts, etc.)
  - Fluid Mixtures: LNG mixtures, binary mixtures

See: Cryogenic Material Properties Database (NIST SRD #152)
http://www.cryogenics.nist.gov/MPropsMAY/materialproperties.htm
NIST Chemistry WebBook (NIST SRD #69)
http://webbook.nist.gov/chemistry/
NIST REFPROP (NIST SRD #23)
http://www.nist.gov/srd/nist23.cfm

LNG Measurement

A User's Manual for Custody Transfer (1985)

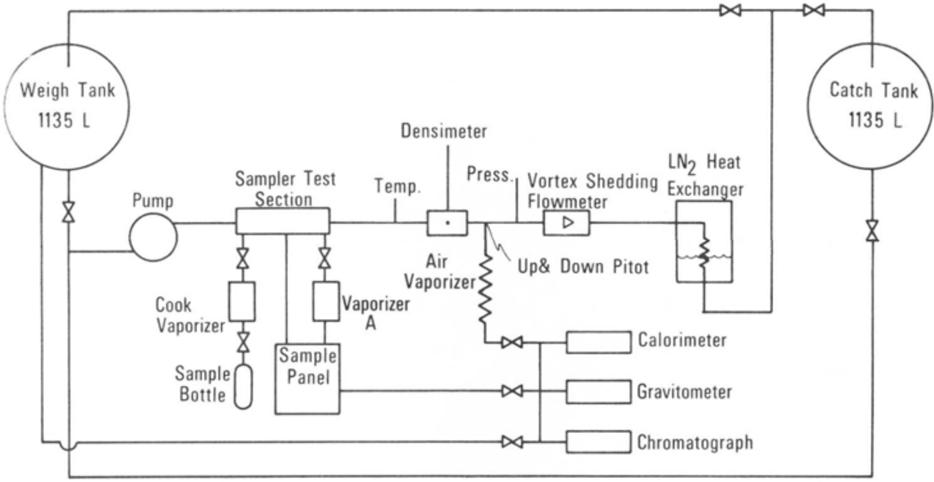
Section 1

- Physical Properties
  - SI System
  - Pure Fluid Properties
  - Combustion Enthalpies

LNG Measurement

- Measurement Elements
  1. Measurement Process Applied to LNG
  2. Sampling and Analysis
     - Sample probe (pitot, side tube, …)
     - Sample conditioner (vaporizing)
     - Gas analyzer (GC, MS, integrator, packings, etc.)
     - Operating parameters/procedures

Laboratory tests
LNG Flow Loop
Shipboard Tests (at Canvey Island, England)
U(3σ, heating value) = 0.3 %



LNG Measurement

- Measurement Elements

3. Calorific Value: *calorimeters, calculations, comparisons*
4. Density Values:

Cryogenic density reference system—*calibrations of LNG densimeter transfer standards U* (LCH4) = 0.055 %

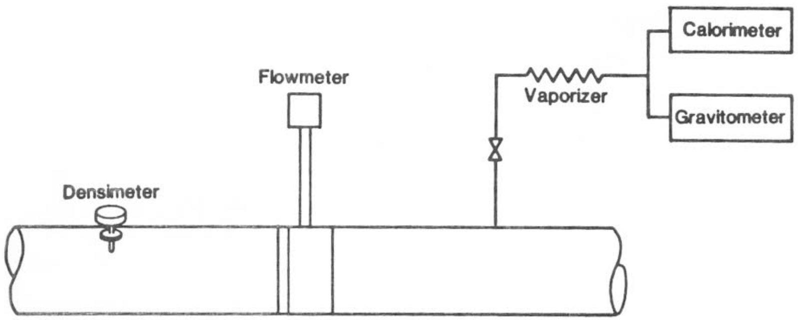
Portable Reference Densimeter—*tests of commercial LNG densimeters*

Experimental LNG Density—*NBS densimeters; pure fluids; binary mixtures, multicomponents* (NBS Monograph 172, 1983)

Mathematical Models for LNG *Densities—ECS, HS, Klosek & McKinley (r), cell*

See: ASTM D4784 – 93 (2010) *Standard Specification for LNG Density Calculation Models*
GERG EOS (2004)
NIST REFPROP (NIST SRD #23) http://www.nist.gov/srd/nist23.cfm

LNG Measurement



- Measurement Elements

5. LNG Volume

- Liquid Level Measurement: *capacitance gauges, cable gages, bubbler gauges*
- Volume of Membrane-Type LNG Ship Tanks *U* = 0.1 %
- Volume of Freestanding Prismatic LNG Ship Tanks *U* = 0.1 %
- Volume of Spherical LNG Ship Tanks
- Volume of Cylindrical LNG Shore Tanks *U* = 0.005 %
- Volume from Totalized Flow Rate Metering
  - LNG Measurement Station: *flowmeter, densimeter, calorimeter, gravitometer*

5 cm to 81 cm (2 in *to* 32 in) flow meter scaleup

LNG Measurement

- Applications
  - Measurement Uncertainties: *volume, gas analysis (0.09 %), calculated LNG density (0.23 %), measured density (0.26 %), calculated calorific value (0.77 %), measured calorific value (0.77 %)*
  - Ship Loading/Unloading (0.2 % to 0.4 % calorific value)
  - Pipeline Metering (0.65 % to 0.95 %)
  - Landbased Storage (0.34 % to 0.65 %)

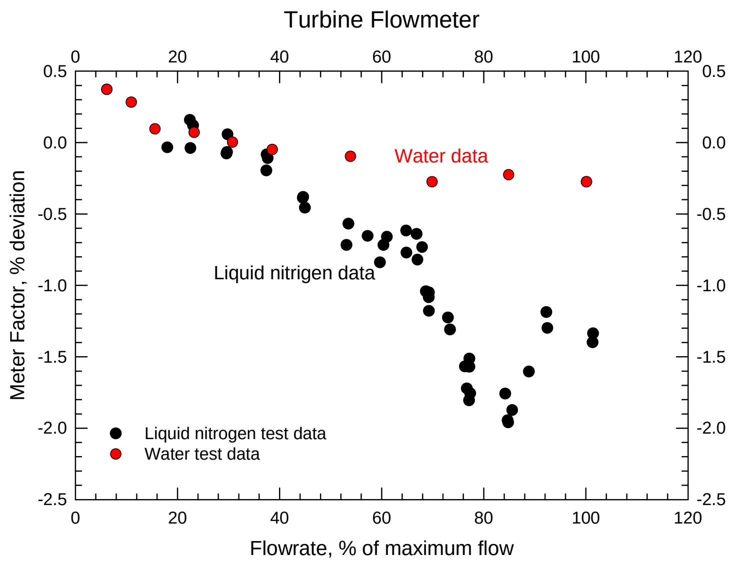
Estimates of Measurement Error
LNG Measurement Process

LNG Measurement Element	Total Uncertainty (percent)	
	Measured	Calculated
Density	± 0.26	± 0.23
Calorific Value	± 0.77	± 0.35

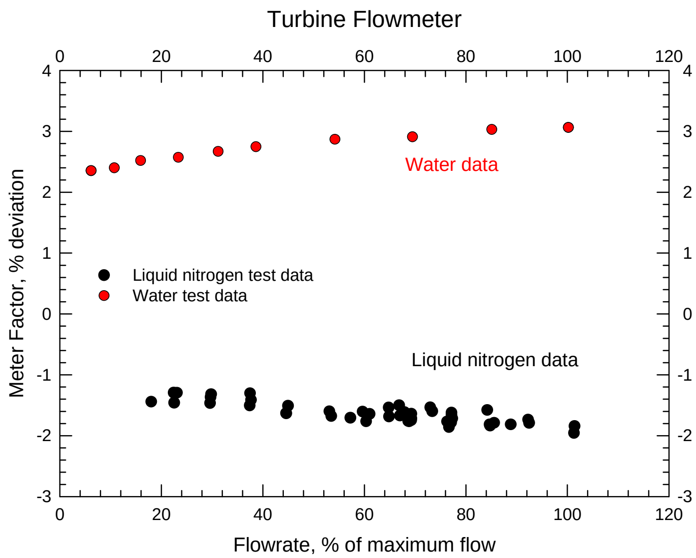
NIST Cryogenic Flow Measurement Facility

Testing and Calibration Services

- Why LN2 Calibrations?



- LN2 vs Water Calibrations



Capabilities

Flow Rate

- Mass : 0.95 to 9.5 kg/s
- Volume : 1.26 to 12.6 L/s

Absolute Pressure

- 0.4 to 0.76 MPa

Temperature

- 80 to 90 K

Metering Characteristics

- Meter Test Section Design
  - 25 to 150 mm diameter
  - 4 meter length
- Flowmeter Calibration Services
  - Turbine
  - Coriolis
  - Ultra Sonic
  - Vortex shedding
- Research Application
  - Prototype meter testing
  - Meter verification at LN2 temperatures
- All measurements are traceable to national standards

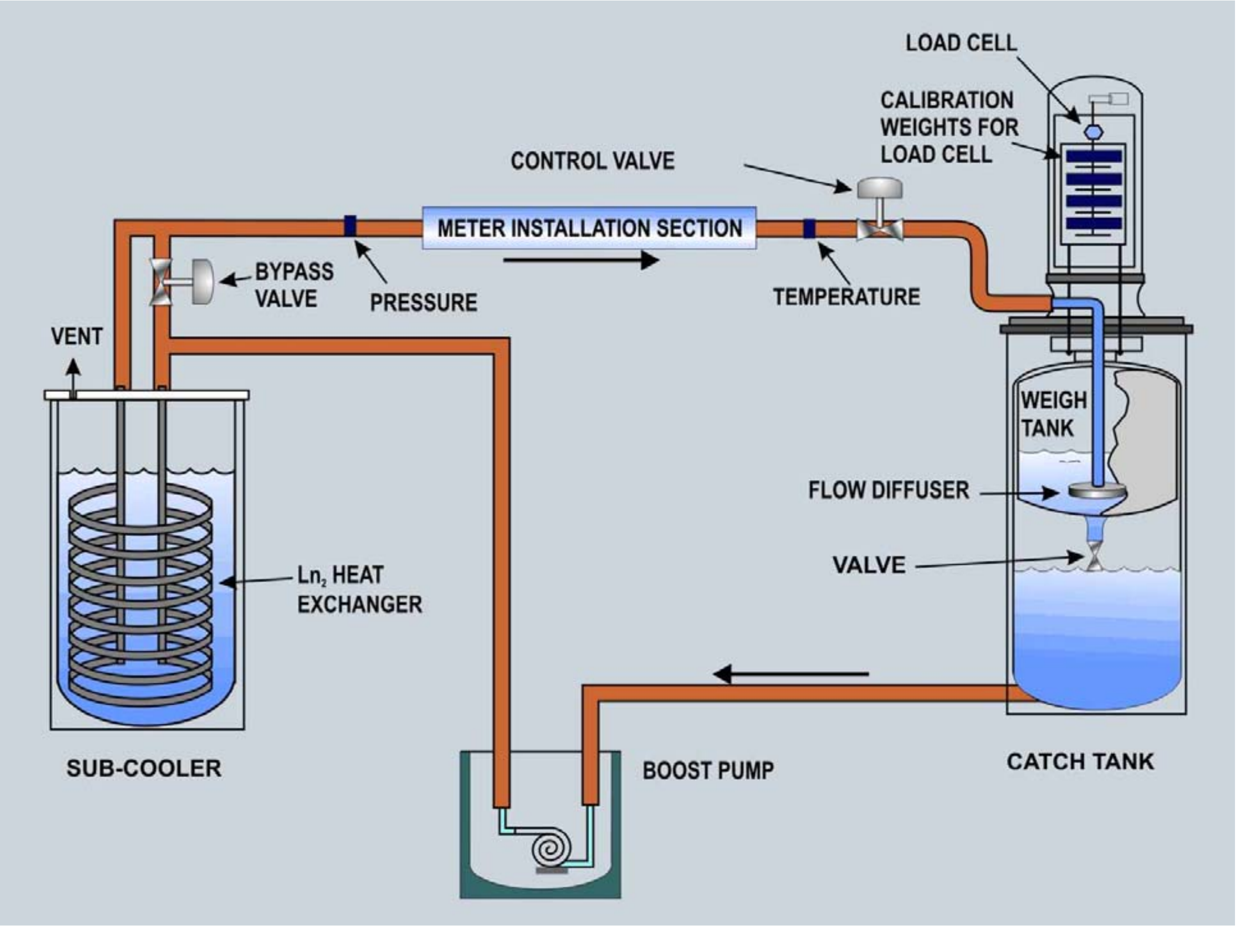
NIST Quality System and Conformance to ISO/IEC 17025

- NIST Quality System in place
- Website: http://www.nist.gov/qualitysystem/
- NIST Quality Assessment
- NIST Assessment Review Board
- NIST Measurement Services Advisory Group (MSAG)
- SIM Regional Metrology Organization
- QSTF: Quality Systems Task Force
- BIPM: Bureau International des Poids et Mesures
- CIPM: International Committee for Weights and Measures/ Comité International des Poids et Mesures
- MRA: Mutual Recognition Agreement
- CMC: Calibration and Measurement Capabilities
- Inclusion in Appendix C *Calibration and Measurement Capabilities*

Facility Components and design

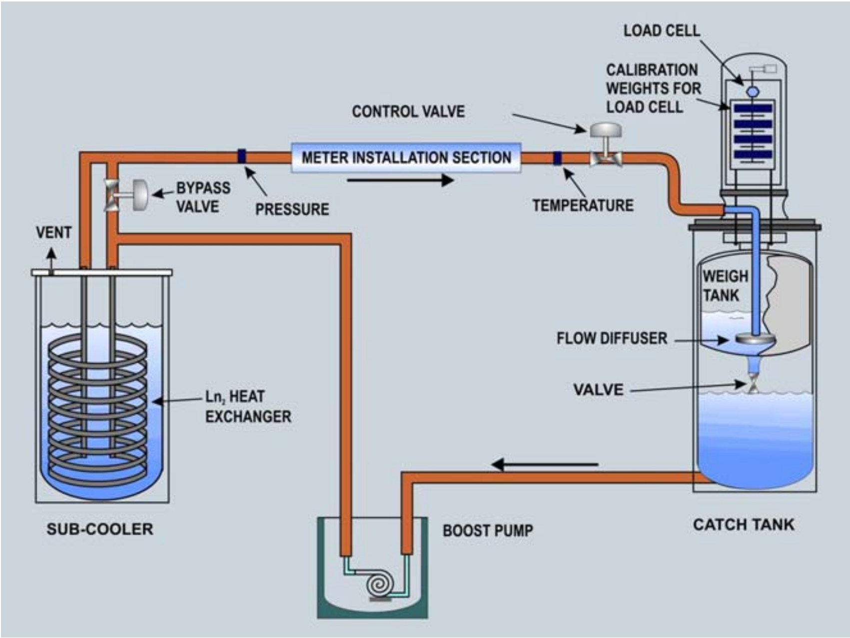
- Liquid nitrogen flow
  - Closed loop
- Mass Measurement
  - Load cell
  - Weigh tank
- Subcooler
  - Remove thermal energy
- Temperature measurement
  - PRT's
- Pressure measurement
  - Pressure transducer

Liquid Nitrogen Flow Facility Schematic



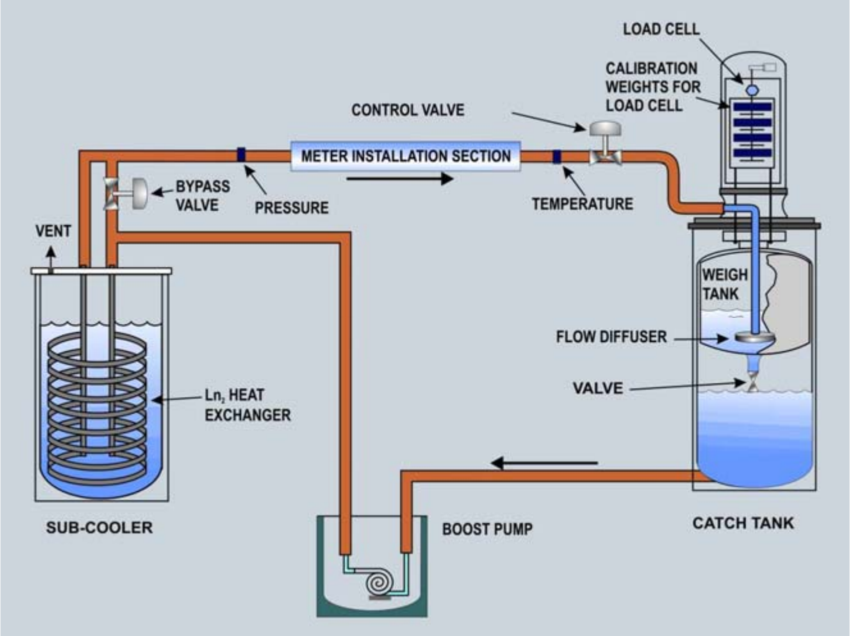
Facility Description

- Liquid nitrogen is the process fluid
- Circulated throughout the closed loop by a variable speed pump
- Flows into a subcooler where thermal energy is removed
  - due to pumping
  - ambient heat leak
- Temperature in the test section controlled by
  - adjusting the subcooler liquid level
  - diverting flow around the subcooler
  - an inline heater
- Pressure in the test section controlled by
  - Downstream control valve
  - Introducing helium gas pressure



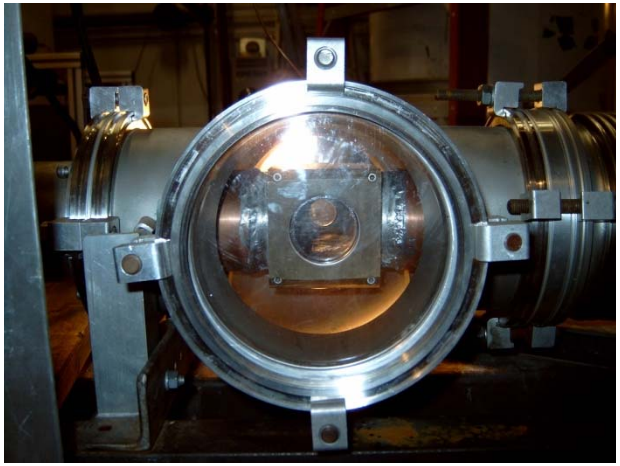
Nitrogen Flow Procedure

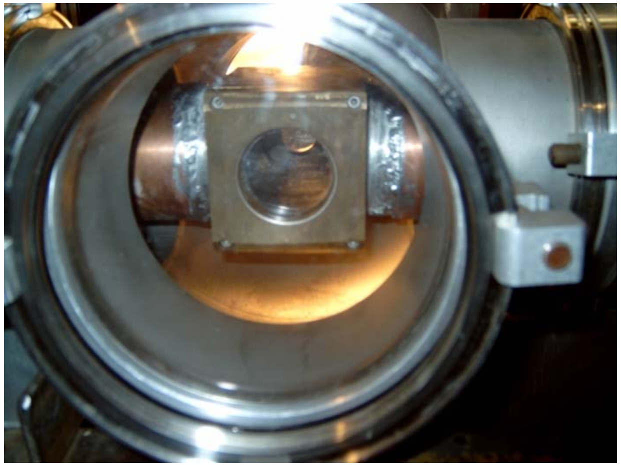
- Catch tank liquid volume creates positive head pressure on centrifugal pump
  - Prevents cavitation and bubbling
- Fluid circulates in 76.2 mm vacuum-jacketed copper pipeline
- Liquid flows into diffuser to remove vertical component of velocity at bottom of aluminum weigh tank
  - 0.378 m^3 capacity
- Liquid flows through valve at bottom of weigh tank and into stainless steel pressure vessel
  - 0.443 m^3 capacity
- When temperature, pressure, and flowrate reach steady-state condition
  - weigh tank valve closed
  - System timers and data acquisition begin
  - Predetermined amount of liquid nitrogen accumulates in weigh tank
  - Varies from approx. 440 kg to 1320 kg
- Mass collected for each data point is determined by
  - Load cell output voltage values
  - Total time the mass was collected



Liquid Nitrogen Flow Stream

- Sapphire window provides ability to observe flow directly downstream of flowmeter test section prior to entering weigh tank
- Internal liquid line is vacuum-jacketed to clearly view flow stream for gas bubbles





Data Acquisition

- Once liquid level reaches preset value, test point begins
- Timer is initiated and data acquisition begins
- Frequency output from flowmeters is input into counters to totalize meter pulses
- Pressure and temperature are measured for density calculations in meter test section as well as in weigh and catch tanks for buoyancy calculations and system monitoring

Uncertainty in Liquid Nitrogen Mass Flow Measurement

Components of the mass flow uncertainty

- Load cell sensitivity measuring the mass of the LN2
  - Weights
  - Voltage
  - Sensitivity equation
  - Pressure
- Buoyancy evaluated for each mass measurement
  - Buoyancy of LN2 accumulated during the data point
  - Buoyancy of the immersed diffuser and pipe in LN2
  - Change in buoyancy of LN2 accumulated before the data point begins
- Time measurement
  - Multi-function datalogger used to measure within 1 ms
- Mass balance between test section and weigh tank
  - Finite volume of LN2 in piping between meter and weigh tank
  - Provided density gradient in pipe remains constant during data point, mass collected in weigh tank is equivalent to mass passing through meter
    - Pressure drop remains nearly constant
    - Temperature gradient less than 70 mK

Total Uncertainties (1σ) for Mass Measurement

Source of Uncertainty:	Type A (%)	Type B (%)	Combined (%)
Load Cell Sensitivity	0.050	0.002	.050
Buoyancy Correction	0.0001	0.068	0.068
Mass between Test Section and Weigh Tank (liquid N2)	0.011		
Total for Mass Measurement	0.051	0.068	0.085
Expanded Uncertainty, k=2			0.170
Time		0.001	0.001
Total for Mass Flow Rate	0.051	0.068	0.085
Expanded Uncertainty, k=2			0.170

Total Uncertainties (1σ) for Volumetric Flow Rate Measurement

Source of Uncertainty in Liquid Volume Flow	Nominal Values: T=85 K Mass = 182 kg	P = 620 kPa Time = 100 s
	Type A (%)	Type B (%)
EOS (REFPROP)		0.02
$\delta\rho/\delta T\|_P$	0.025	0.013
$\delta\rho/\delta P\|_T$ ($P_{Test\ Section}$, Baro)	0.0002	0.0002
Uncert. in mass flow rate	0.051	0.068
Total (in quad)	0.057	0.072
Total (Type A + B, in quad), k=2		0.184

Calibration Procedure

- Statistical calibration program developed
  - Uses fractional factorial test plan
  - Provides ability to separate main effects and low-order interactions from one another
- Allows evaluation of meter for sensitivity to
  - Pressure
  - Temperature
  - Flowrate
  - Thermal cycling
- 60 data points taken over two-day period
  - 5 temperatures
  - 12 flowrates
  - 5 pressures

Data Analysis

For volumetric flow rate, predicted performance of meter in measuring flow rate is compared to flow rate determined by facility:

Meter Mass: $M_R = P\rho / K$ (1)

Percent Deviation $= (M_R - M_{NIST}) * 100 / M_{NIST}$ (2)

Where,

- M_R = mass calculated from the flowmeter output
- P = flowmeter output in total pulses
- ρ = liquid density determined from the flow facility temperature and pressure measurements
- K = meter factor in pulses per unit volume
- $M_{NIST =}$ mass determined by the flow facility

For mass flow rate, only second equation is used

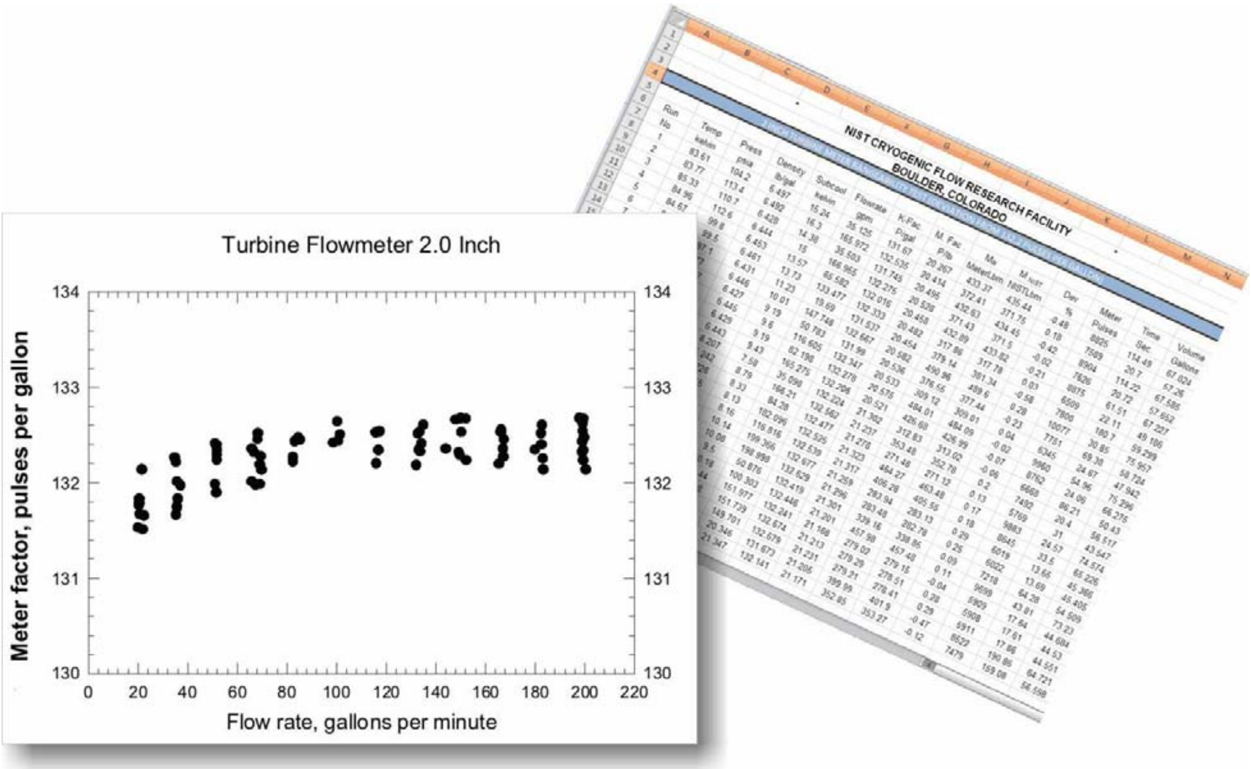
Data Analysis

The deviation data are fitted by least-squares method to mathematical model equation:

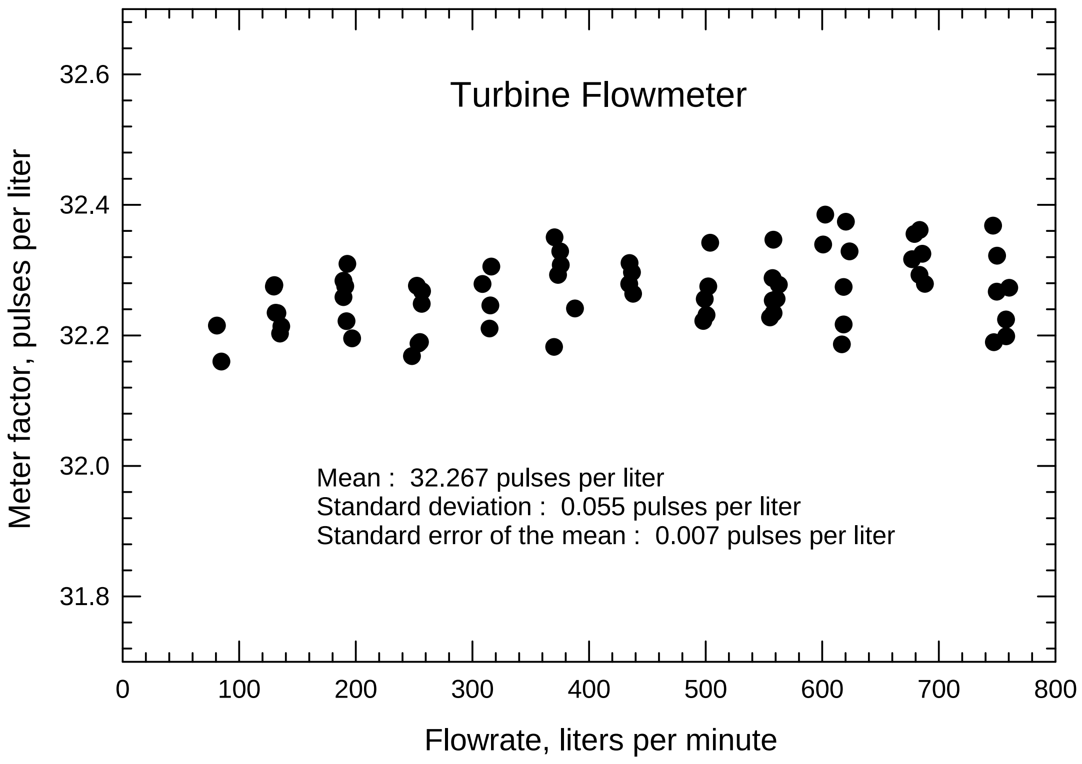
$$Dev(\%) = A + BQ + CQ^2 + DQ^3 + ET + FP$$

- Dev(%) = percent deviation
- T = fluid temperature
- Q = volume flowrate
- P = liquid nitrogen pressure
- A,B,C,D,E,F = coefficients

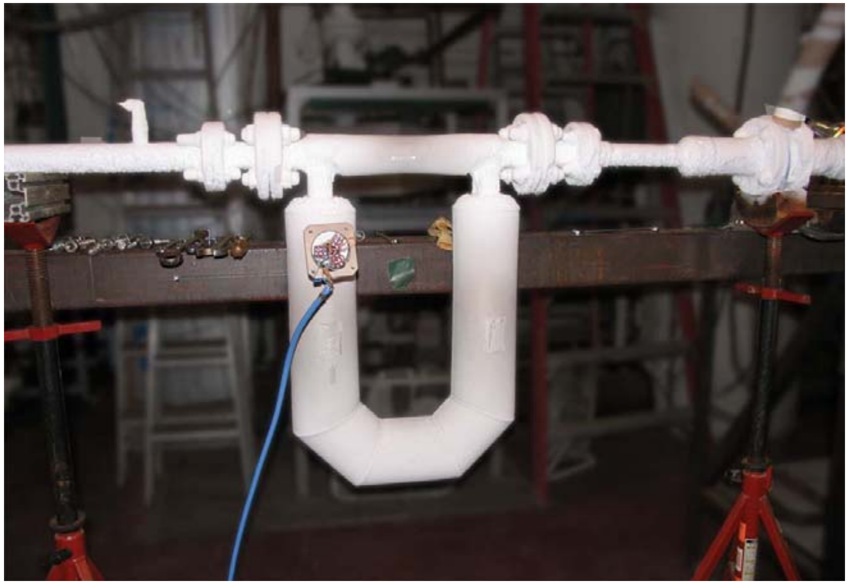
Sample Data Results



Typical Meter Calibration with Statistical Summary

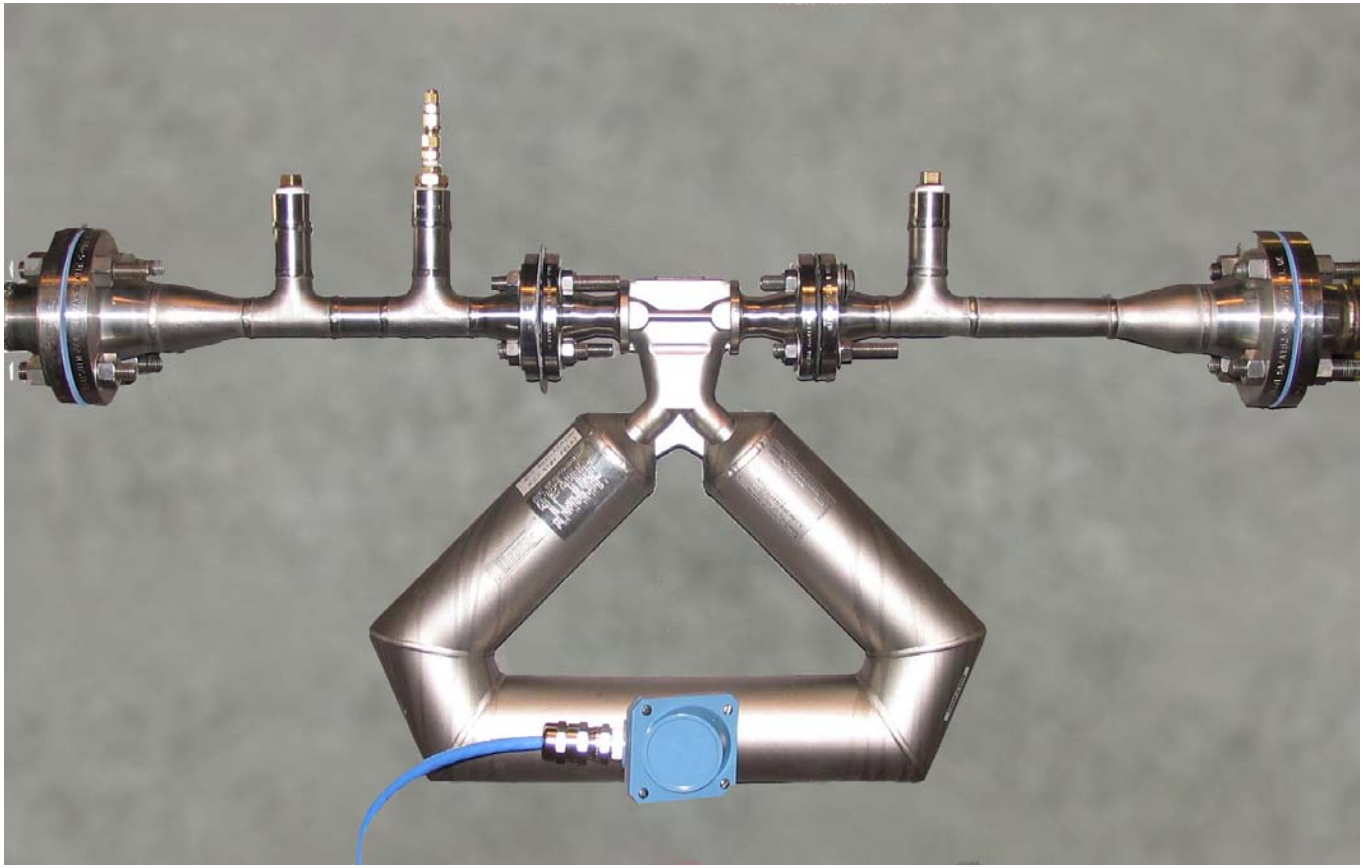


38 mm Meter Installation



50 mm Flowmeter Installation

25 mm Flowmeter Installation



150 mm Flowmeter Installation

Summary

- NIST Cryogenic Flow Facility is well instrumented in test section and weigh tank/catch tank assembly
- A meter evaluation test plan is used to determine sensitivity to pressure, temperature, flowrate, and thermal cycling
- Combined expanded uncertainties (k =2) for totalized mass and volume cryogenic flow measurement are 0.17% and 0.18% respectively
- Facility has been well used in evaluating various types of flowmetering devices to determine their suitability for measuring cryogenic flows
- It is anticipated that LNG and LH2 will have expanding roles as alternative fuels, and NIST will be able to provide assistance in developing accurate measurement methods for cryogenic liquid flows

Thank you for your attention