CHAPTER C8

CRYOGENIC PIPING SYSTEMS

INTRODUCTION

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Cryogenics (from the Greek "kryo-genikos," meaning cold generation) is the science and technology associated with very low temperatures. Depending on one's point of view, any temperature below −20°F can be set to establish such a demarcation. Here the −20°F point has been selected because it normally represents the onset of embrittlement for ordinary carbon steels in typical structural applications.

Cryogenics is not a separate branch of physics, since it obeys all laws of ordinary physics. In fact, cryogenics is low-temperature physics. The reasons for its special treatment, therefore, are not because of its uniqueness as a science but rather because of the very special problems it creates as a technology. These problems relate to embrittlement of materials, large displacements (expansion and contraction), rapid change of phase due to large heat fluxes (big delta T), and small latent heats of the fluids involved.

In order to obtain a better appreciation of the special considerations involved in cryogenic piping system applications, it was felt that it would be necessary to review the behavior of materials at cryogenic temperatures and the physical and thermodynamic properties of cryogenic fluids. These considerations are covered in the sections "Properties of Cryogenic Fluids" and "Materials Used in Cryogenic Piping Systems." Additionally, cryogenic piping system design is discussed in the sections "Piping Systems Design—Fluids" and "Piping Systems Design—Mechanical."

From the strictly heuristic point of view of fundamental applications of scientific principles there are hardly any differences between cold box piping and all other types. Nevertheless, we are making a special topic of cold box piping because of the confined spaces involved and the conceptual arrangements required to satisfy logistically workable and economically feasible process considerations. Such piping is discussed in the section "Cold Box Piping."

The coverage on cryogenic distribution systems, as provided under the sections "Liquid Storage and Conversion Systems" and "Mobile Equipment System," considers more than just piping; it covers the functional design philosophy of cryogenic fluid storage and distribution and provides quite an insight into the logistics of the entire operation.

With the advent of chip making, the need for ultra-high-purity inert gases has come into clear focus, and industry has responded to this need by developing suitable storage and distribution systems. These aspects of cryogenic piping systems are discussed in the last section.

Naturally, the drive behind most technologies is economic in nature, and in this respect cryogenics is no exception. This is certainly much more so when it comes to liquid distribution because there is no other motive. Industrial gases can certainly be distributed in the compressed gaseous form, even in bulk quantities, if costs are not a consideration. Such economic aspects are discussed in the next section.

References for each section are at the end of each section.

ECONOMIC PARAMETERS OF CRYOGENIC FLUID DISTRIBUTION SYSTEMS

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Cryogenic processes are typically applied to commodity chemicals that exist as gases at normal ambients. Such gases are liquefied at reduced temperatures and are normally maintained at saturated conditions. The processing techniques generally involve both the liquid and gaseous phases and exploit the dramatic changes in physical and thermodynamic properties that occur with changes of state. Cryogenic temperatures are applied at each step in the process of bringing these gases to the final consumer, including production, distribution, and storage.

Typically, industrial gases are found in mixtures in which some of the components have commercial value. Two of the most important examples of such mixtures are gases from certain hydrocarbon wells and the Earth's atmosphere. The feed streams are separated by liquefaction and subsequent fractional distillation with the produced streams delivered in either the gaseous or liquid state. Cryogenic distillation allows a wide choice in the degree of separation, ranging from crude to extremely fine. It is a highly efficient process with power consumed chiefly in refrigeration lost to the environment and pressure lost in the product streams. Economic considerations in the liquefaction and distillation process involve trade-offs between operating efficiency and capital expenditure. No other method is as versatile or as effective as cryogenic separation of industrial gas mixtures for commodity usage.

The key to industrial gas distribution and storage operations is the use of a cost-effective method that increases the product density. The transport or storage vessel is reduced to manageable dimensions in this way. The historical solution to this problem is elevated pressures at ambient temperatures. Indeed, high-pressure cylinders and receivers made to a variety of U.S. Department of Transportation (USDOT) and ASME specifications are widely used today to store gases at pressures exceeding 2000 psig (138 bar). These vessels are typically of single-piece forged construction involving fabrication and inspection procedures that enable them to safely operate at ultimate to design stress ratios of 3:1. In spite of these measures to achieve an efficient package, the ratio of vessel weight to product weight is extremely high. Therefore, the cost of storing and transporting these gases in such vessels is very high relative to the value of the product they contain. Some of the characteristics of typical high-pressure receivers and cylinders are summarized in Table C8.1a and Table C8.1b.

TABLE C8.1a Typical Specifications for Seamless Forged Pressure Vessels
ASME Pressure Vessel Code. Sizes, capacities, and design pressures

Dimensions									
OD	in	16	16	20	20	24	24	24	24
	mm	406	406	508	508	610	610	610	610
Min wall*	in	1.25	1.25	1.303	1.303	0.817	0.817	1.154	1.154
	mm	31.8	31.8	33.1	33.1	20.8	20.8	29.3	29.3
Max length	ft	30	30	23	23	24	24	20	20
	mm	9144	9144	7010	7010	7315	7315	6069	6096
Max water volume	cu ft	27	27	34.2	34.2	60.2	60.2	46.1	46.1
	liter	765	765	969	969	1705	1705	1306	1306
Material class†		65	70	65	70	65	70	65	70
Weight									
Unit weight	lb/ft	222.4	222.4	294.4	294.4	230.1	230.1	311.3	311.3
	kg/m	331	331	438.1	438.1	343.9	343.9	463.3	463.3
Max vessel weight	lb	6672	6672	6670	6770	5537	5537	6241	6241
	kg	3026	3026	3071	3071	2516	2516	2831	2831
Design pressure									
SF = 3	psi	5833	6667	4811	5500	2450	2800	3500	4000
	bar	402	460	332	379	169	193	241	276
SF = 4	psi	4383	5000	3615	4124	1841	2100	2630	3000
	bar	302	345	249	284	127	145	181	207

* Nominal wall = min wall ÷ .875.
† Material is SA 372 Grade J.
Source: CP Industries, Inc.

TABLE C8.1b Typical Specifications for Seamless Forged Pressure Vessels
USDOT specification. Dimensions and data for typical vessel sizes.

USDOT Specification[1]							
	psi	3AA-2400	3AAX-2400	3AAX-2900	3AAX-3855	3T-2400	3T-2850
	bar	3AA-166	3AAX-166	3AAX-200	3AAX-266	73T-166	3T-197
Dimensions							
OD	in	24	22	22	18	22	22
	mm	610	559	559	457	559	559
Min wall	in	0.584	0.536	0.647	0.705	0.415	0.492
	mm	14.8	13.6	16.4	17.9	10.5	12.5
Max length[2]	ft-in	6–11.25	40	36	40	40	40
	m	2.11	12.19	10.97	12.19	12.19	12.19
Max average	lb	1168	5616	6056	5935	4455	5162
weight[3]	kg	530	2547	2747	2692	2021	2341
Nom water	cu ft	15.7	91	79.6	56.8	93.3	91.8
volume	liter	445	2577	2254	1609	2642	2600

[1] Specifications are as defined in United States Code of Federal Regulations Title 49.
[2] Length can be varied to meet specific requirements.
[3] Includes end fittings.
Source: CP Industries, Inc.

Liquefaction is another approach that has been widely used for efficient storage and transportation of industrial gases. Gases typically emerge from the liquefaction process saturated at approximately atmospheric pressure and from this point are transported and stored in cryogenic vessels. These are typically double-walled vessels with an inner container designed for the working pressure and temperature of the product and an outer casing designed for ambient temperatures and external pressure. In between the two vessels is a high-performance insulation system which is usually evacuated for the purpose of further enhancing thermal protection. Structural members to support the inner container and piping to provide access to it are also located in the insulation space. Heat is continuously entering the vessel through the insulation, supports, and internal piping. This heat will make the liquid contents boil, and the resultant gas must be removed from the tank if the pressure and temperature of the contents are to be held steady. For this reason, heat leak must be minimized. The thermal efficiency of the tank design is expressed in terms of the percentage of full capacity that will be lost per day when the tank is held at atmospheric pressure. This parameter is termed the *normal evaporation rate* (NER) and is product specific. Table C8.2 summarizes the specifications of typical tankage applied in various production and distribution functions.

Gaseous state storage at high pressures and liquid state storage at cryogenic temperatures both have their place in industry today. Therefore, it is important to compare their relative economics. The primary costs to be compared are those for storage vessel construction, transportation, power to achieve the storage state, and product loss. The construction methods used in forged high-pressure receivers are very different from those used in high thermal efficiency cryogenic temperature vessels. However, the resultant costs per unit weight may be considered equal for

TABLE C8.2 Typical Specifications for Cryogenic Storage Vessels

Model		DM-900	DM-1500	DM-3000	DM-6000	DM-9000	DM-11000	DM-13000
Capacity								
Warm Water	gal	960	1585	3100	6020	9180	11300	13300
	liter	3634	6000	11734	22788	34749	42771	50345
Net Liquid	gal	900	1490	3000	5880	8900	11000	13000
	liter	3407	5640	11356	22258	33689	41638	49209
Oxygen	1000 cu ft	104.0	171.0	345.3	676.8	1024.4	1266.0	1496.0
	cu m	2734	4497	9076	17790	26926	33277	39322
Nitrogen	1000 cu ft	83.8	138.4	279.3	547.5	828.7	1024.0	1210.0
	cu m	2203	3638	7341	14391	21782	26916	31805
Argon	1000 cu ft	101.2	167.6	337.5	661.5	1001.3	1237.0	1462.0
	cu m	2660	4405	8871	17388	26319	32515	38429
Dimensions								
Height	ft-in	15-9	15-9	15-10	26-2	29-7	31-5	36-0
	m	4.8	4.8	4.83	8.0	9.0	9.6	11.0
Diameter	ft-in	6-6	6-6	8-0	8-0	9-6	10-2	10-2
	m	1.9	1.9	2.4	2.4	2.9	3.1	3.1
Weight								
Tare	1000 lb	9.5	10.5	15.5	27.9	37.0	47.0	50.0
	1000 kg	4.3	4.8	7.0	12.7	16.8	21.3	22.7
Oxygen	1000 lb	18.1	24.8	44.1	83.9	121.8	152.0	174.0
	1000 kg	8.2	11.2	20.0	38.1	55.2	68.9	78.9
Nitrogen	1000 lb	15.6	20.6	35.7	67.7	97.0	121.0	138.0
	1000 kg	7.1	9.3	16.2	30.7	43.9	54.9	62.6
Argon	1000 lb	20.0	28.0	50.4	96.3	140.5	175.0	201.0
	1000 kg	9.1	12.7	22.9	42.3	63.7	79.4	91.2
Design parameters								
NER (oxygen % per day)		0.4	0.4	0.5	0.3	0.26	0.25	0.23
MAWP	psig	250	250	250	250	250	250	250
	bar	17.2	17.2	17.2	17.2	17.2	17.2	17.2

Source: Taylor-Wharton Div Harsco Corp.

TABLE C8.3 Weight Ratio: Lading/Vessel

Cryogenic storage vessel						
Volume		MAWP		Lading weight/Tare weight		
gal	liters	psig	bar	N2	O2	AR
900	3407	250	17.2	0.64	0.90	1.10
1490	5640	250	17.2	0.96	1.35	1.65
3000	11356	250	17.2	1.31	1.84	2.25
5880	22258	250	17.2	1.42	2.00	2.44
8900	33689	250	17.2	1.62	2.29	2.80
11000	41638	250	17.2	1.58	2.23	2.72
13000	49209	250	17.2	1.75	2.48	3.02
Gas receiver						
Volume		MAWP		Lading weight/Tare weight		
cu ft	liters	psig	bar	N2	O2	AR
60.2	1705	2800	193	0.14	0.18	0.23
46.1	1306	4000	276	0.13	0.17	0.21
34.2	969	5500	379	0.11	0.15	0.19
27.0	765	6667	460	0.10	0.14	0.17

Source: Praxair, Inc.

rough comparison. Therefore, storage vessel and transportation costs for alternative systems can be compared by the ratio of the vessel weight to product weight. Table C8.3 compares this ratio for several products stored in typical gas and liquid storage vessels. Generally the total cost (capital plus operating) to liquefy a product is lower than that required to compress it as a gas to receiver pressure. Indeed, receivers are most often charged with gas pumped to pressure in the liquid state and subsequently heated to ambient temperature rather than by gas state compression. Product losses to be expected in various liquid systems can be estimated from the NER specifications given in Table C8.2.

Considering these circumstances, cryogenic liquid phase storage is the most economical approach in most cases. Gas storage should be considered where the requirement involves small quantities or long periods of nonuse or difficulty in disposing of the gas boil-off expected in a cryogenic system.

The piping used in cryogenic systems obviously must meet the structural demands imposed by low temperatures. From an economic point of view, the thermal efficiency of the piping system must be carefully considered since the heat of addition to the system will ordinarily result in loss of product. There are two important factors of product loss involved in piping systems that must be considered: refrigeration required to bring the line to operating temperature (cool down) and steady state heat addition. Table C8.4 gives these parameters for uninsulated lines, lines insulated with closed-cell polyurethane foam, and lines insulated with radiation shields in high vacuum (vacuum insulation).

TABLE C8.4 Heat Addition[a]: Steady State and Cooldown

Steady state heat addition								
	Nominal pipe size NPS (DN)							
	1 (25)		2 (50)		3 (80)		4 (100)	
Insulation	Btu /hr-ft	watt/m	Btu /hr-ft	watt/m	Btu /hr-ft	watt/m	Btu /hr-ft	watt/m
Uninsulated[b]	283.0	271.9	533.0	512.2	787.0	756.2	1037.0	996.4
Polyurethane foam[c] thickness								
1 in (25.4 mm)	16.0	15.4	25.3	24.3	32.9	31.6	41.3	39.7
2 in (50.8 mm)	10.8	10.4	15.4	14.8	19.8	19.0	24.1	23.2
3 in (76.2 mm)	8.8	8.5	12.2	11.7	15.2	14.6	18.2	17.5
Vacuum insulation[d]	0.5	0.4	0.8	0.8	1.0	1.0	1.2	1.2
Cooldown								
	Nominal pipe size NPS (DN)							
	1 (25)		2 (50)		3 (80)		4 (100)	
Insulation	Btu /ft	watt-hr/m	Btu /ft	watt-hr/m	Btu /ft	watt-hr/m	Btu /ft	watt-hr/m
CU tube[e]	20.2	19.4	54.0	51.9	103.0	99.0	166.0	159.5
SS pipe[f]	28.3	27.2	52.3	50.3	98.5	94.6	127.4	122.4
Polyurethane foam[g] thickness								
1 in (25.4 mm)	2.5	2.4	3.7	3.6	4.9	4.7	7.9	7.6
2 in (50.8 mm)	7.3	7.0	9.6	9.2	12.2	11.7	16.5	15.9
3 in (76.2 mm)	14.5	13.9	18.0	17.3	22.0	21.1	28.0	26.9
Vacuum insulation[g]	7.1	6.8	12.9	12.4	21.6	20.8	26.6	25.6

[a] Liquid Nitrogen Service.
[b] 8-mph wind over frosted insulated line.
[c] Closed-cell polyurethane foam with PVC cover.
[d] Evaculated laminar radiation shields.
[e] ASTM B-88 Type L.
[f] Schedule 5.
[g] Heat addition due to insulation only.
Source: Praxair, Inc.

PROPERTIES OF CRYOGENIC FLUIDS

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Physical and thermodynamic properties of cryogenic fluids constitute important data that are needed for the design of cryogenic piping systems. The following discussion, tables, figures, and references are furnished with this need in mind.

Transport property data are readily available for the more common pure cryogenic fluids. The bibliography at the end of this section lists sources of physical properties and thermodynamic tables and charts, which supply the detailed data required for the design of piping systems. Table C8.5M (Metric) summarizes some of the more important properties for a number of cryogenic fluids.

Thermodynamic charts show pressure-temperature-phase-density (or specific volume) and enthalpy (heat content, H) relationships for a specific fluid under a variety of formats. Entropy data are often included but are not generally required for the purposes under consideration in this chapter.

Fig. C8.1 is a pressure-enthalpy chart for nitrogen, which will be used to illustrate the behavior of fluids in transport systems, and various uses of pressure-temperature-volume-enthalpy (P-T-V-H) data. The most obvious use may be to determine the density (the reciprocal of specific volume) of a fluid which is being transported under constant pressure and temperature conditions. Referring to the chart, for example, the density of nitrogen at 101.5 psia (0.7 MPa) and 80°F (300°K) is found to be approximately 0.5 lb/ft^3 (8.0 kg/m^3). Other uses of the chart are illustrated in the examples which follow.

The dome-shaped curve at the bottom left of the chart encloses the two-phase (vapor-liquid) region. Nitrogen at pressure and temperature conditions to the left of the dome is a *saturated or subcooled liquid*. Conditions to the right of the dome correspond to *saturated or superheated vapor*. Vapor and liquid phases coexist within the dome at a unique pressure for a specific temperature. This pressure is designated as the *vapor pressure* of the fluid at that temperature. The length of the isobar (constant pressure) line between the sides of the dome is proportional to the heat input which is required to completely vaporize the fluid at the pressure and corresponding temperature. The fraction of the fluid which is vapor at a condition corresponding to a specific point along the isobar is equivalent to the fraction of its distance from the saturated liquid line, divided by the total length of the isobar within the dome.

The point at the very peak of the dome represents a unique condition of pressure and temperature which is designated as the *critical point* of the fluid. As nitrogen approaches 493 psia (3.40 MPa) and −232.5°F (126.2°K) from any direction, all distinctions between the characteristics of vapor and liquid phases disappear. Liquid being warmed at a pressure above the critical pressure behaves as a dense fluid, which gradually approaches the characteristics of a high-pressure gas at higher temperatures, without passing through any observable phase change.

The vertical lines on a pressure-enthalpy chart correspond to a constant heat content condition. Movement along these lines designates an isenthalpic or adiabatic (no heat input or output) process. Flow through reasonably short or insulated runs of piping or fittings, or at a temperature close to that of the environment, approaches isenthalpic behavior.

When a fluid flowing within a pipe is throttled through a valve, the change in its pressure-temperature-phase and density relationships is essentially isenthalpic. If a fluid is initially a gas which is reduced in pressure, its decrease in pressure and density may be accompanied by a change in temperature (Joule-Thomson effect). In most cases this will be a decrease in temperature, as shown, for example, by nitrogen at 725 psia (5 MPa) and −153.7°F (170°K) following the 60.23 Btu/lb(m) (140kJ/kg) isenthalp to about −215°F (136°K) when throttled to atmospheric pressure. At conditions more remote from the critical point, the temperature change is less dramatic. A rise in temperature may occur when the fluid is well above its critical temperature. This is shown by nitrogen at 80°F (300°K) when throttled to 5800 psia (40 MPa) from a higher pressure, but it is more typically encountered with hydrogen or helium under normal processing conditions.

When a saturated liquid is throttled to lower pressure, an isenthalpic line is followed into the dome, indicating partial vaporization of the downstream fluid. For example, if liquid nitrogen which is initially saturated at 290 psia (2 MPa) and −251.6°F (115.58°K) is throttled to atmospheric pressure, the downstream condition will be about 45 percent vapor. This will be accompanied by a change in density from about 35.77 lb(m)/ft^3 to 0.637 lb(m)/ft^3 (573 to 10.2 kg/m^3), or a factor of 56 increase in volume. If nitrogen vapor is throttled from a point above the dome, it may partially liquefy (retrograde condensation) or, within a very narrow range of conditions, pass through the two-phase region before ending up as all vapor at a final low-pressure condition.

The large change in specific volume which may occur with throttling—and particularly vaporization—may necessitate a substantial increase in the diameter of downstream piping in order to maintain reasonable velocities of the fluid. If the process leads to a two-phase downstream condition, there may be some slippage between the phases (i.e., the relative fraction of the vapor and liquid inventoried in the piping may differ from that of the net throughput). Two-phase flow characteristics can be extremely complex, and determination of this behavior is beyond the scope of this discussion.

An important consideration in the design of a cryogenic piping system arises from recognition that a confined fluid cannot increase in specific volume, or change in density, when heated. Extreme overpressurization can result when a cryogenic liquid which is trapped between valves is warmed, for example by heat leak. Confined nitrogen which is initially liquid at 101.5 psia (0.7 MPa) will [following the 43.70 lb(m)/ft^3 (700 kg/m^3) constant density line] exceed 1160 psia (8 MPa) when warmed to −270.7°F (105°K) and approach 14,500 psia (100 MPa) at −100°F (200°K). This makes overpressure protection mandatory wherever entrapment of a cryogenic liquid or (initially) high-pressure cold gas is possible.

Pressure-enthalpy charts for other cryogenic fluids are similar to those for nitrogen. Charts for oxygen and argon are provided as Figs. C8.2 and C8.3, respectively. Sources of charts for additional fluids are given in the bibliography at the end of this section. The thermodynamic property interrelationships of these fluids are similar to those which have been illustrated for nitrogen.

Rapid exposure of a partially confined cryogenic liquid to heat may result in overpressurization even when some outlet is provided. The possibility of a cryogenic liquid spill into a warmer environment must be considered in designing any enclosure around a piping system.

TABLE C8.5M (Metric) Physical and Thermodynamic Properties of Cryogenic Fluids

	Helium	Hydrogen equil	Hydrogen normal	Neon	Nitrogen	Carbon Monoxide	Air	Fluorine	Argon	Oxygen	Methane	Krypton	Nitric Oxide
Formula	He	e-H2	n-H2	Ne	N2	CO	Mixture	F2	Ar	O2	CH4	Kr	NO
Molecular weight	4	2.02	2.02	20.18	28.01	28.01	28.96	38	39.95	32	16.04	83.80	30.01
Triple point													
Temperature, K		13.83	13.95	24.55	63.15	68.15		53.48	83.81	54.36	90.68	115.76	109.51
Pressure, kPa		7.0	7.2	43.3	12.5	15.4		0.252	69.1	0.15	11.7	73.2	21.9
Heat of fusion, J/g		58.2	58.1	16.26	25.74	30.0		24.2	29.58	13.9	58.6	19.6	76.6
Normal boiling point,													
Temperature, K	4.22	20.27	20.38	27.09	77.35	81.7	78.7/81.7	84.95	87.29	90.19	111.64	119.78	121.38
Density, kg/m^3													
Liquid	124.9	70.8	70.7	1,205	805.4	789	875.4	1524	1,394	1,134	423	2,414	1,269
Vapor	16.89	1.34	1.329	9.58	4.60	4.40	4.51	5.64	5.77	4.49	1.818	8.8	3.04
Heat of vaporization, J/g	20.4	442	448	85.8	199.7	215.8	201.1	174.5	160.78	212.1	510	107.6	459
Specific heat, J/g-K													
Liquid	4.52	9.71	9.75	1.91	2.042	2.15	1.966	1.511	1.07	1.737	3.43	0.55	
Vapor	9.08	12.23	12.20	1.16	1.34	1.22	1.130	0.825	0.56	0.971	2.15	0.26	
Viscosity, g/m-s													
Liquid	0.0036	0.0133	0.0133	0.13	0.17	0.17	0.18	0.237	0.27	0.189	0.12	0.38	
Vapor	0.0012	0.0011	0.0011	0.0042	0.0052	0.0056	0.01	0.0072	0.0070	0.0074	0	0.01	0.01
Thermal conductivity, W/m-K													
Liquid	0.026	0.119	0.119	0.114	0.14	0.140	0.14	0.16	0.12	0.150	0.193	0.090	
Vapor	0.009	0.017	0.017	0.01	0.0070	0.0069	0.01	0.01	0.0057	0.0076	0.01	0	0.01
Critical point													
Temperature, K	5.19	32.98	33.25	44.40	126.2	132.85	132.5	144.3	150.65	154.58	190.55	209.39	180
Pressure, kPa	227.5	1,293	1,297	2,653	3,400	3,494	3,766	5,215	4,898	5,043	4,599	5,496	6,480
Density, kg/m^3	69.64	31.4	31	483.1	313.1	303.9	316.5	574	535.7	436.2	162.7	910.7	517.4
Gas at 101.325 kPa, 294.26 K													
Density, kg/m^3	0.17	0.08	0.08	0.836	1.160	1.161	1.2	1.57	1.66	1.33	0.665	3.48	1.244
Specific heat, J/g-K	5.19	14.84	14.29	1.030	1.041	1.039	1.01	0.83	0.52	0.92	2.226	0.25	0.971
Specific heat ratio	1.67	1.383	1.407	1.67	1.401	1.402	1.4	1.36	1.67	1.4	1.31	1.67	1.40
Viscosity, g/m-s	0.020	0.0089	0.0089	0.031	0.0174	0.0176	0.0183	0.02	0.02	0.0204	0.01	0.03	0.02
Thermal conductivity, W/m-K	0.15	0.190	0.183	0.048	0.0254	0.0247	0.0261	0.03	0.02	0.0263	0.033	0.01	0.026

TABLE C8.5M (Metric) Physical and Thermodynamic Properties of Cryogenic Fluids (*Continued*)

	R-14	Ozone	Xenon	Ethylene	Ethane	Nitrous Oxide	Hydrogen Oxide	Acetylene	Carbon Dioxide	Hydrogen Sulfide	Propylene	Propane	Ammonia
Formula	CF4	O3	Xe	C2H4	C2H6	N2O	HCl	C2H2	CO2	H2S	C3H6	C3H8	NH3
Molecular weight	88.01	48	131.3	28.05	30.07	44.01	36.46	26.04	44.01	34.08	42.08	44.1	17.03
Triple point													
Temperature, K	89.52	80.65	161.36	103.97	89.9	182.33	158.97	192.1	216.58	187.62	87.89	85.47	195.41
Pressure, kPa	0.11	0	81.60	0.12	0	87.9	14	120.0	518	23.2	2E-07	3E-07	6.1
Heat of fusion, J/g	7.95		17.26	119	95.1	149	54.9	144	204.9	69.7	71.4	79.9	332
Normal boiling point,													
Temperature, K	145.09	161.85	165.03	169.38	184.57	184.67	188.15	189.1s	194.67s	212.81	225.46	231.08	239.72
Density, kg/m^3													
Liquid	1633	1354	2941	567.8	544	1233	1190			965	609	581	682
Vapor	7.74	3.70	10.00	2.09	2.056	3.11	2.5	2.10		2.0	2.36	2.42	0.89
Heat of vaporization, J/g	134.1	295.8	95.73	481.4	491	376	443	818s	573s	548	439	428	1,371
Specific heat, J/g-K													
Liquid	0.91		0.35	2.41	2.43		1.61			1.83	2.03	2.246	4.43
Vapor	0.51		0.17	1.37	1.40		0.85			1.02	1.31	1.46	2.24
Viscosity, g/m-s													
Liquid	0.32		0.49	0.164	0.168		0.407			0.42	0.176	0.199	0.262
Vapor	0.01		0.01	0.0058	0.0059	0.01	0.0090			0.01	0.01	0.0064	0.0081
Thermal conductivity, W/m-K													
Liquid	0.09	0.22	0.07	0.19	0.15	0.17	0.34			0.23	0.149	0.129	0.587
Vapor	0.01		0.0033	0.0077	0.0093	0.01	0.01		0.01	0.01	0.0093	0.0114	0.0175
Critical point													
Temperature, K	227.6	261.1	289.74	282.35	305.42	309.6	324.69	308.32	304.12	373.2	365.57	369.8	405.5
Pressure, kPa	3740	5570	5,821	5,042	4,880	7,255	8310	6,139	7,374	8937	4,665	4,240	11,353
Density, kg/m^3	629	537	1,100	214.2	204.6	454	450	231.2	467.8	346	223.4	220.5	235.2
Gas at 101.325 kPa, 294.26 K													
Density, kg/m^3	3.66	1.99	5.47	1.169	1.256	1.836	1.52	1.086	1.832	1.43	1.77	1.861	0.713
Specific heat, J/g-K	0.690	0.825	0.160	1.525	1.737	0.88	0.8	1.67	0.839	1.02	1.52	1.67	2.09
Specific heat ratio	1.16		1.68	1.248	1.20	1.30	1.41	1.26	1.316	1.32	1.21	1.14	1.32
Viscosity, g/m-s	0.017		0.023	0.010	0.0092	0.01	0.01	0.01	0.015	0.0126	0.01	0.01	0.0101
Thermal conductivity, W/m-K	0.0155		0.01	0.020	0.020	0.017	0.014	0.02	0.0159	0.018	0.02	0.017	0.023

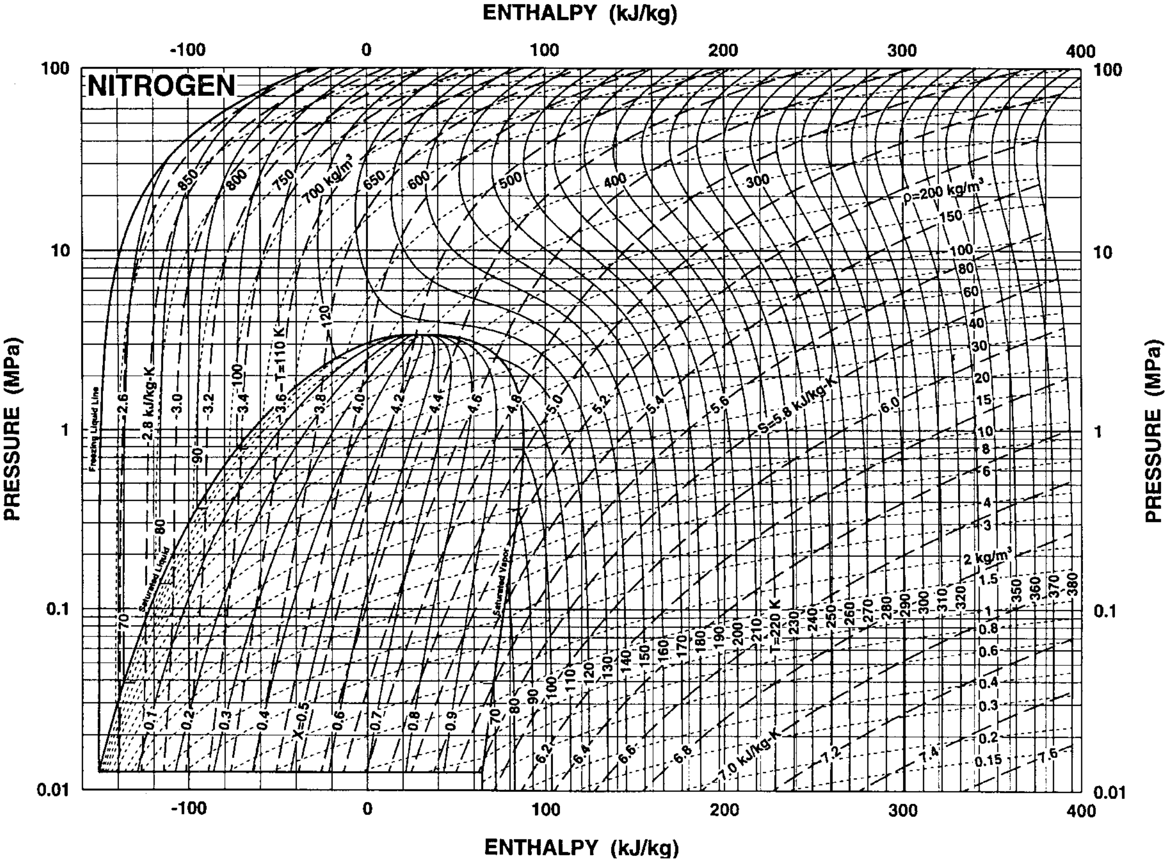

FIGURE C8.1 *P-H* diagram for nitrogen (P = 0.01–100 MPa). (*Reproduced with permission from Jacobsen, Penoncello and Lemmon*, Thermodynamic Properties of Cryogenic Fluids, *Plenum Press, NY, 1997.*)

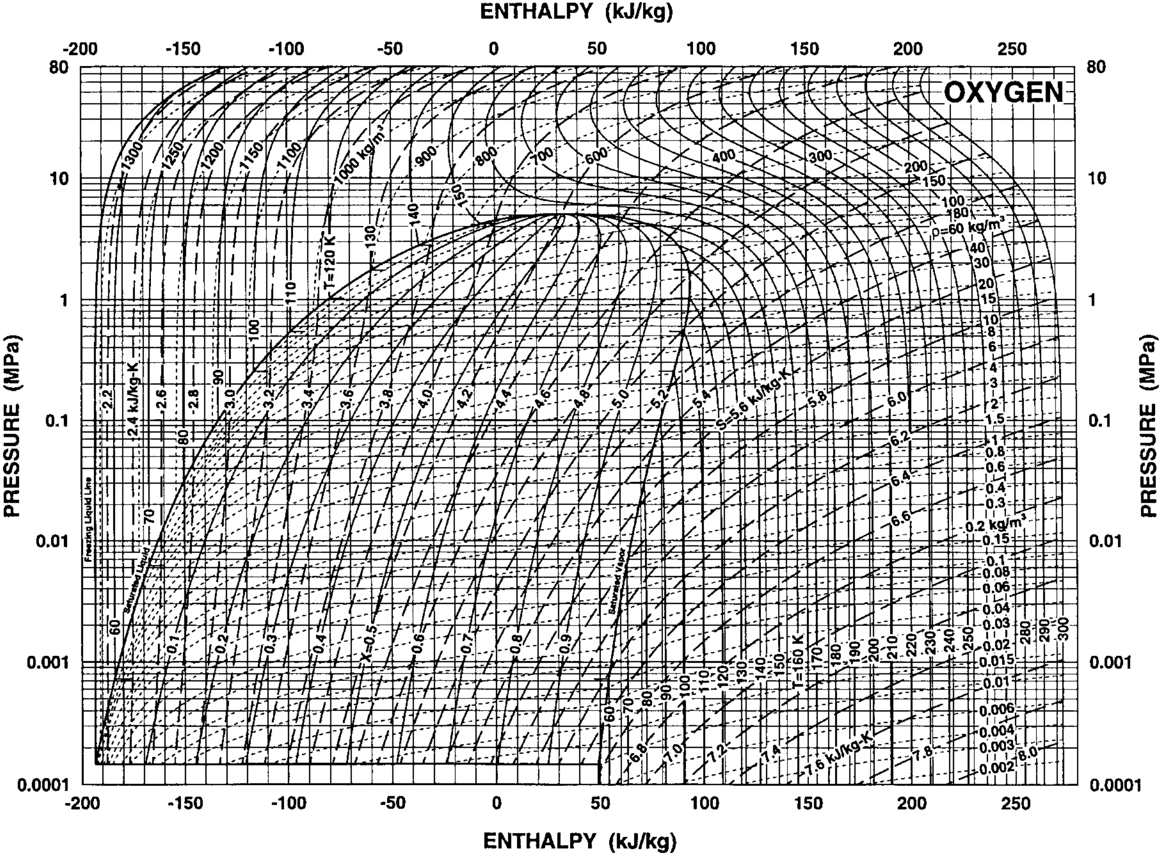

FIGURE C8.2 *P-H* diagram for oxygen (P = 0.0001–80 MPa). (*Reproduced with permission from Jacobsen, Penoncello and Lemmon*, Thermodynamic Properties of Cryogenic Fluids, *Plenum Press, NY, 1997.*)

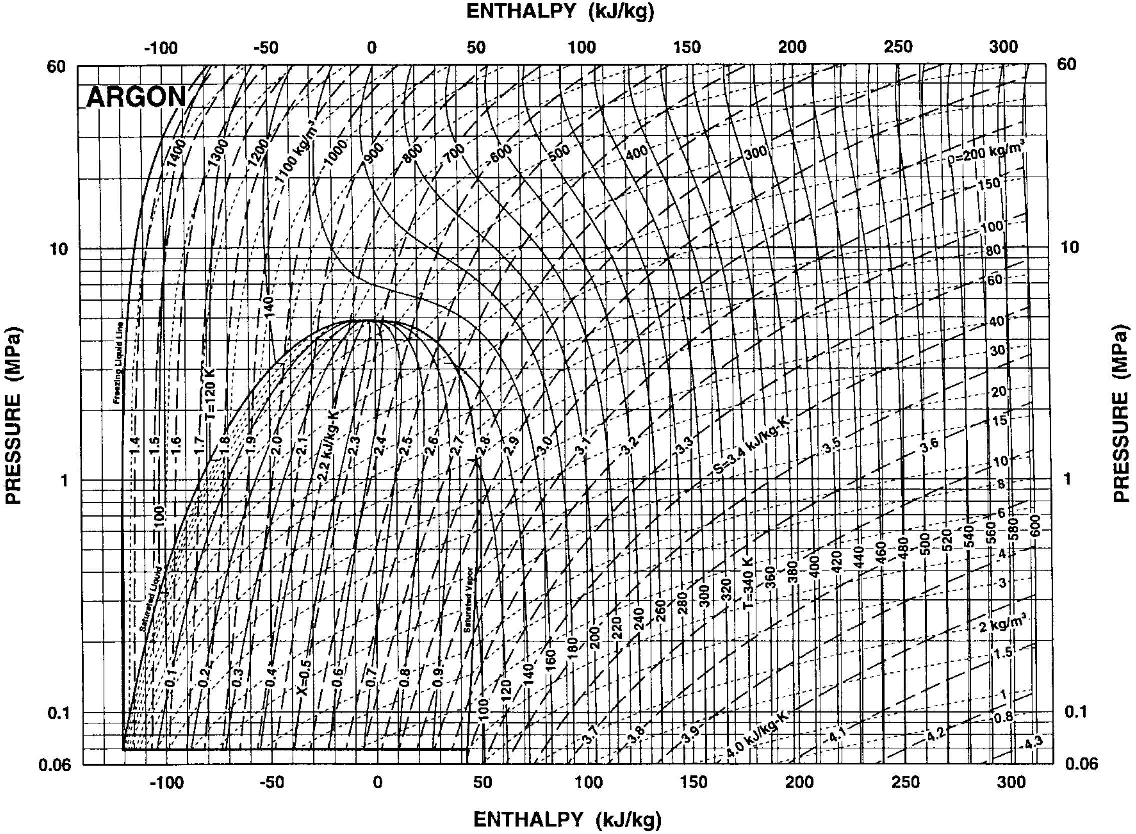

FIGURE C8.3 *P-H* diagram for argon ($P = 0.06–60$ MPa). (*Reproduced with permission from Jacobsen, Penoncello and Lemmon,* Thermodynamic Properties of Cryogenic Fluids, *Plenum Press, NY, 1997.*)

Certain fluids require special consideration. Liquid carbon dioxide, for example, is not stable at atmospheric pressure. (This is indicated on Table C8.5M by a triple-point pressure above atmospheric). Loss of pressure on a liquid carbon dioxide system (or a high-pressure gas system at sufficiently low temperature) will result in dry ice formation.

Physical and thermodynamic property data on cryogenic mixtures are not readily available in easily applied chart or tabular format (as they are for pure fluids). Required design data are usually computer generated from complex correlations for specific cases. Knowledge of pure fluid behavior will, however, provide an awareness of many factors which require consideration in the design of a cryogenic piping system.

Among the specific differences between the behavior of pure fluids and that of mixtures is a potentially much broader range of temperature and pressure conditions within the two-phase region for the latter case, and differences in composition between the individual phases and that of the overall mixture. There is also a possibility of one of the components freezing under conditions wherein other components may exist as gas and/or liquid mixtures. Although a component which is below its normal freezing point may have considerable solubility in the liquid or gas mixture, the possibility of solid formation should be anticipated when any of the components is present in a mixture at a temperature below its triple point. This situation may result from the mixing of two streams, neither of which contains a frozen component.

The designer should have knowledge of the combustion and physiological properties of the fluids being handled. Air and other oxidants must be excluded from piping transporting flammable fluids and from any enclosure (such as a cold box) into which leakage may occur. Conditions which may concentrate flammable contaminants present a danger in air separation. A number of cryogenic fluids are toxic, and all can present an asphyxiation hazard, particularly within confined areas. Venting of these fluids must take into account the possibility that temperature differences from the ambient air may lead to unexpected localized concentration buildups which threaten the safety of personnel and equipment.

BIBLIOGRAPHY

Sources of Physical and Thermodynamic Properties of Cryogenic Fluids:

AGA Gas Handbook, AGA AB, Lindingo, Sweden, 1985.

ASHRAE Thermodynamic Properties of Refrigerants, American Society of Heating, Refrigerating and Air Conditioning Engineers, Atlanta, 1986. Tabular data and charts.

Beaton, C.F. and G.F. Hewitt, *Physical Property Data for the Design Engineer,* Hemisphere Publishing, New York, 1989.

CRC Handbook of Chemistry and Physics, CRC Press, Boca Raton, FL.

Jacobsen, R.T., S.G. Penoncello, and E.W. Lemmon, *Thermodynamic Properties of Cryogenic Fluids,* Plenum Press, New York, 1997. Extensive tables and charts for the major cryogens.

Lange's Handbook of Chemistry, McGraw-Hill, New York.

Matheson Gas Data Book, Matheson, Lyndhurst, NJ, 1980. Includes data for specialty gases, including toxicity, reactivity (flammability, corrosiveness, incompatibility, etc.) and safe handling procedures. *Matheson Unabridged Gas Data Book;* Matheson, East Rutherford, NJ, 1975. More extensive property data for these components.

Maxwell, J.B., *Data Book on Hydrocarbons,* D. Van Nostrand, Princeton, NJ, 1950. Tabular and chart data for light hydrocarbons.

National Standard Reference Data Service of the USSR: A Series of Property Tables, Hemisphere Publishing, Washington, 1987. Comprehensive thermodynamic and physical property tables in volumes covering helium, nitrogen, methane, ethane, oxygen, air, ethylene, halocarbon refrigerants, and rare gases (neon, argon, krypton and xenon).

Perry, R.H. and D.W. Green, *Chemical Engineers' Handbook,* sixth ed., McGraw-Hill, New York, 1984. Tabular data and thermodynamic charts for many cryogens.

Raznjevic, Kuzman, *Handbook of Thermodynamic Tables and Charts,* Hemisphere Publishing, Washington, 1976.

Reid, R.C., J.M. Prausnitz, and T.K. Sherwood, *The Properties of Gases and Liquids,* third ed., McGraw-Hill, New York, 1977. Estimating methods for physical properties of pure fluids and mixtures.

Tables of Thermodynamic and Transport Properties of Air, Argon, Carbon Dioxide, Carbon Monoxide, Hydrogen, Nitrogen, Oxygen, and Steam (originally issued as NBS Circular 564), Pergamon Press, New York, 1960.

TRC Thermodynamic Tables—Hydrocarbons, 1992. *TRC Thermodynamic Tables—Non-Hydrocarbons,* 1993. Thermodynamic Research Center, The Texas A&M University System, College Station, TX. Multiple volumes.

MATERIALS USED IN CRYOGENIC PIPING SYSTEMS

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Important considerations in the selection of materials for cryogenic piping systems include suitable mechanical and physical properties, compatibility with process fluids, fabricability, cost, and compliance with regulatory codes.

The subject of materials for cryogenic applications has been generously treated in the technical literature. For those interested in additional background information, a few selected works in this area appear in the references and bibliography at the end of this section. The prime focus of this section, however, is cryogenic piping for the chemical process industry and commercial cryogenic distribution applications. Consequently, this overview will be deliberately limited in its scope and coverage, and many materials (such as those used in aerospace applications) will not be covered. The materials that will be covered include ferrous alloys, nonferrous alloys, and nonmetallic materials.

FERROUS MATERIALS

Ferrous alloys most often encountered in cryogenic piping applications are usually classified as ferritic or austenitic types. The terms *austenitic* and *ferritic* refer to the predominant crystallographic phases ferrite or austenitic, which are body centered cubic (BCC) and face centered cubic (FCC), respectively.

Ferritic Alloys

Most of the steels in common use are ferritic. This classification also covers steels which are martensitic as a result of heat treatment. Low cost, ease of fabricability, and high strength via heat treatment are major reasons for their popularity. Ferritic alloys, however, can exhibit a ductile-to-brittle toughness transition. The Charpy Impact Test, by which measurements of energy absorption, lateral expansion, and ductile fracture appearance are made, is the *most* common method of measuring this transition.

However, the ductile-to-brittle transition temperature in ferritic steels can be influenced by a number of variables involved in the steel-making process which control the levels of residual elements, inclusion shape, and heat treatment techniques which in turn control grain size and crystallographic morphology. A comprehensive review of these approaches is beyond the scope of this document. However, alloying with nickel, reduction of sulphur levels, and reductions in grain size may be stated as the most popular approaches with ferritic alloys. Table C8.6 contains a listing of some of the most common ferritic alloy steels used in cryogenic piping. Minimum service temperatures are included as well as the applicable ASME specifications.

Table C8.7 and Table C8.7M (Metric) contain a listing of the same materials as

TABLE C8.6 Typical Ferrous Alloys Used in Cryogenic Piping

Alloy	Minimum temperature*	ASME[3] specification	Comment
C-Mn steel[1]	−46°C (−50°F)	SA-333[3] Grade 1	Aluminum killed, fine grain practice
2¼% Ni steel[1]	−73°C (−100°F)	SA-333[3] Grade 7	Aluminum killed, fine grain practice
3½% Ni steel[1]	−101°C (−150°F)	SA-333[3] Grade 3	Aluminum killed, fine grain practice
9% Ni steel[1]	−196°C (−320°F)	SA-333[3] Grade 8	Aluminum killed, fine grain practice
304 Stainless steel[2]	−254°C (−425°F)	SA-312[3]	
304L Stainless steel[2]	−254°C (−425°F)	SA-312[3]	
316 Stainless steel[2]	−196°C (−320°F)	SA-312[3]	
316L Stainless steel[2]	−196°C (−320°F)	SA-312[3]	
347 Stainless steel[2]	−254°C (−425°F)	SA-312[3]	

* Design minimum temperature for which material is normally suited without impact testing other than that required by material specification.

[1] Ferritic steels.

[2] Austenitic steel.

[3] Corresponding ASTM specifications are A333 and A312.

Source: Praxair, Inc.

TABLE C8.7 Typical Mechanical and Physical Properties of Ferrous Alloys

Alloy	ASME[3] spec.	Temperature[4] (°F)	Ultimate tensile strength (ksi)	0.2% Offset Yield strength (ksi)	Elongation in 2 in. (%)	Charpy impact strength (ft-lb.)	Thermal expansion (in/in °F × 10⁻⁶)	Thermal conductivity (BTU(hr · ft · °F)
C-Mn steel[1]	SA 333 Grade 1	RT −50	55	30	21(1)	70 50	6.5 3.0	30 —
2¼% Ni steel[2]	SA 333 Grade 7	RT −100	65 75	35 40	18(1)	58 20	— —	— —
3½% Ni steel[2]	SA 333 Grade 3	RT −150	100 120	75 87	18(1)	96 22	5.8 4.8	21 —
9% Ni steel[2]	SA 333 Grade 8	RT −320	115 170	90 135	25 27	47 25	5.8 4.8	15.7 7.6
304 stainless steel	SA 312 TP 304	RT −425	85 250	38 70	45 3	115 75	9.0 1.2	9 0
304L stainless steel	SA 312 TP 304L	RT −425	80 225	37 65	45 31	60 60	Same as 304 Same as 304	Same as 304 Same as 304
316 stainless steel	SA 312 TP 316	RT −325	87 197	38 65	45 56	— —	9.0 7.8	9 0
316L stainless steel	SA 312 TP 316L	RT −325	85	38	45	—	Same as 316 Same as 316	Same as 316 Same as 316
347 stainless steel	SA 312 TP 347	RT −425	90 230	65 70	50 38	60 45	8.7 7.2	8.5 0

* Minimum value as stated in ASME Specification SA-333.

[1] Normalized, aluminum killed, fine grain practice.

[2] Quenched and tempered.

[3] Corresponding ASTM specifications are A333 and A312.

[4] RT is room temperature.

Source: Praxair Inc.

TABLE C8.7M (Metric) Typical Mechanical and Physical Properties of Ferrous Alloys

Alloy	ASME[3] spec.	Temperature[4] (°C)	Ultimate tensile strength (MPa)	0.2% Offset yield strength (MPa)	% Elong. in 5.1 cm (%)	Charpy impact strength (Joules)	Thermal expansion ($m/m/K \times 10^{-6}$)	Thermal conductivity (W/m · K)
C-Mn steel[1]	SA 333 Grade 1	RT −46	379	207	21(1)	95 68	11.7 5.4	52 —
2¼% Ni steel[2]	SA 333 Grade 7	RT −73	448 517	241 276	18(1)	79 27	— —	— —
3½% Ni steel[2]	SA 333 Grade 3	RT −101	689 11379	517 586	18(1)	130 30	10.4 8.6	36 —
9% Ni steel[2]	SA 333 Grade 8	RT −196	793 1172	621 931	25 27	64 34	10.4 8.6	27 13
304 Stainless steel	SA 312 TP 304	RT −254	586 1724	262 483	45 3	156 102	16.2 2.1	15 0
304L Stainless steel	SA 312 TP 304L	RT −254	552 1551	255 469	45 31	81 81	Same as 304 Same as 304	Same as 304 Same as 304
316 Stainless steel	SA 312 TP 316	RT −198	600 1358	262 448	45 56	— —	16.2 12.4	15 0
316L Stainless steel	SA 312 TP 316L	RT −198	586	262	45	—	Same as 316 Same as 316	Same as 316 Same as 316
347 Stainless steel	SA 312 TP 347	RT −254	621 1586	469 483	50 38	81 61	15.6 12.9	14 0

* Minimum value as stated in ASME Specification SA-333.

[1] Normalized, aluminum killed, fine grain practice.

[2] Quenched and tempered.

[3] ASTM specifications are A333 and A312.

[4] RT is room temperature.

Source: Praxair Inc.

Table C8.6 with typical data, including some data not found in the ASME specifications. Included in Tables C8.7 and Table C8.7M are mechanical properties such as strength, impact, and elongation, as well as thermodynamic data such as thermal expansion and thermal conductivity at cryogenic temperatures (boiling point at 1 atmosphere).

Austenitic Alloy Steels

In addition to providing data on the ferritic alloy steels, Tables C8.6, Table C8.7, and Table C8.7M also contain a listing of austenitic stainless steels which are likely to be encountered in cryogenic piping. Most of the austenitic alloy steels used in cryogenic piping are chromium-nickel stainless steels of the AISI 300 type, such as 304, 304L, 316, and 316L. Other stainless steels classified as martensitic, duplex, and precipitation hardening also exist; however, the preceding alloys are most commonly used in cryogenic piping for chemical process and distribution applications.

A major consideration in the use of the 300 Series stainless steels is the improvement in toughness properties they provide and elimination of the sharp ductile-to-brittle transition found in ferritic steels. These alloys have been used to contain and distribute liquid hydrogen and helium. Consequently, while the austenitic stainless steels were originally developed for corrosion resistance, their toughness and excellent fracture properties are what led to their selection for cryogenic piping applications.

Of the 300 Series alloys, the AISI 304 composition is the most popular as measured by tonnage. It should be noted that there is a preference for AISI 316L in the electronics industry for the distribution of high-purity gases that are free of particulates. Furthermore, piping and tubing used in this application are frequently electro-polished, and the trend is for an increase in electro-polishing for ultra-high-purity applications.

Cast versions of the common austenitic stainless steels may be used if valve applications exist. CF3, CF3M, CF8, and CF8M are cast equivalents of 304L, 316L, 304, and 316, respectively. Note that the cast stainless steels have been optimized for castability, and increased delta ferrite levels may adversely affect toughness at cryogenic temperatures. Similar effects may be observed in austenitic weld filler metals, and both castings and weld fillers should be well characterized and qualified prior to use in cryogenic piping systems.

NONFERROUS ALLOYS

Nonferrous alloys encountered in cryogenic piping are usually of the aluminum, cuprous, or nickel families. None of the three alloy families exhibit ductile-to-brittle toughness transition due to the FCC crystal lattice. Common nonferrous alloys used in cryogenic piping appear in Table C8.8, Table C8.9, and Table C8.9M (Metric).

Aluminum Alloys

Common aluminum alloys used in cryogenic process piping do not represent the gamut of what is available in aluminum alloys. The high-strength aluminum alloys

TABLE C8.8 Typical Nonferrous Alloys Used in Cryogenic Piping

Alloy	Tempers	Minimum temperature	ASME[1] spec.
1100 Aluminum	O, H11	−254°C (−452°F)	SB 210
3003 Aluminum	O, H112	−254°C (−452°F)	SB 210
5052 Aluminum	O, H32	−254°C (−452°F)	SB 210
5083 Aluminum	O, H112	−254°C (−452°F)	SB 210
5086 Aluminum	O, H112	−254°C (−452°F)	SB 210
6061 Aluminum	T6	−254°C (−452°F)	SB 210
Copper (C10200, C12200)	Annealed	−198°C (−325°F)	SB 75
Copper-nickel (70600)	Annealed	−198°C (−325°F)	SB 467
Copper-nickel (C71500)	Annealed	−198°C (−325°F)	SB 467
Monel 400, Al-Cu alloy	Annealed	−198°C (−325°F)	SB 165

* Design minimum temperature for which material is normally suitable without impact testing other than that required by material specification.

[1] Corresponding ASTM specifications are B75, B165, B210 and B467.

Source: Praxair Inc.

commonly used in aerospace applications are not used in the chemical process industry because of the lack of ASME Code coverage or other considerations such as corrosion.

Aluminum compositions of the 5083 and 6061 types constitute the highest-strength alloys used in cryogenic applications, and tempers of these alloys are suitable at temperatures as low as −452°F (−254°C). From the toughness standpoint, 5083 would be preferred. However, prolonged exposure to temperatures as high as 152°F (66°C) during thaws can result in both a reduction in corrosion resistance and toughness. For low-strength applications, the 3000 series are also used.

Copper and Cuprous Alloys

Most of the early work in the cryogenic industry was accomplished through the use of copper process vessels, piping, and tubing. Aluminum has extensively replaced copper and cuprous alloys in the fabrication or air separation plants. However, copper and cuprous piping are still extensively used in piping and tubing runs from cryogenic tankage for several reasons which include ease of fabrication (e.g., brazing, soldering), resistance to ignition, and combustion resistance in oxygen-enriched systems. It may be noted that orbital arc-welded stainless piping systems are increasingly replacing cuprous piping for the delivery of particulate and contaminant-free electronic grade gases.

TABLE C8.9 Typical Mechanical and Physical Properties of Nonferrous Alloys*

Alloy	ASME[1] Spec.	Temp. (°F)	Ultimate tensile strength (ksi)	0.2% Offset yield strength (ksi)	Elongation in 2 in. (%)	Charpy impact strength (ft · lb)	Thermal expansion (in/in °F × 10^{-6})	Thermal conductivity [BTU(h · ft · °F)]
1100 Aluminum 0, H112	SB-210	RT −452	13S 24H 24S	5S 152H 8S	40S 10H 56S	16S 70H	13 5	125 160
3003 Aluminum 0, H112	SB-210	RT −452	16S 29H 32S	6S 186H 9S	35S 7H 48S	16S 70H	12 5	92 85
5052 Aluminum O, H32	SB-210	RT −452	43S 46H 76S 86H	37S 290H 47S 379H	12S 8H 42S 30H		13.2	75 7
5083 Aluminum 0, H1112	SB-210	RT −452	42S 44H 63S	21S 193H 23S	22S 16H 32S		13 5	68 4
5086 Aluminum 0, H112	SB-210	RT −452	38S 42H 78S 96H	17S 117H 20S 179H	22S 12H 38S 30H		13.2	73
6061 Aluminum T6	SB-210	RT −452	45 70	40 58	12 25	10 12	10 2	99
Copper (C10200, C1200)	SB-75	RT −452	33 52	10 14	45 69	56 75	9.5 5.0	150 75
90/10 Cu-Ni CDA 706	SB-467	RT −325	44	16	42		9.5	26
70/30 Cu-Ni CDA 715	SB-467	RT −325	44 85	20 31	40 60	79 87	9	17
Monel 400	SB-165	RT −325	80 115	25 50	42 64	56 50	7.5 2.5	15 5

* The letters S and H designate soft and hardened conditions respectively.

[1] Corresponding ASTM specifications are B75, B165, B210 and B467.

Source: Praxair, Inc.

TABLE C8.9M (Metric) Typical Mechanical and Physical Properties of Nonferrous Alloys*

Alloy	ASME[1] Spec.	Temp. (°C)	Ultimate tensile strength (MPa)	0.2% Offset yield strength (MPa)	Elongation in 5.1 cm (%)	Charpy impact strength (Joules)	Thermal expansion (m/m/K × 10^{-6})	Thermal conductivity (W/(m·K))
1100 Aluminum 0, H112	SB-210	RT	90S 165H	34S 152H	40S 10H	22S 95H	23	216
		−254	165S	55S	56S		9	450
3003 Aluminum 0, H112	SB-210	RT	110S 200H	41S 186H	35S 7H	22S 95H	21	159
		−254	221S	62S	48S		9	147
5052 Aluminum 0, H32	SB-210	RT	296S 317H	255S 290H	12S 8H		23	130
		−254	524S 593H	324S 379H	42S 30H			12
5083 Aluminum 0, H1112	SB-210	RT	290S 303H	145S 193H	22S 16H		23	117
		−254	434S	159S	32S		9	7
5086 Aluminum 0, H112	SB-210	RT	262S 290H	117S 117H	22S 12H		23	126
		−254	538S 662H	138S 179H	38S 30H			
6061 Aluminum T6	SB-210	RT	310	276	12	14	18	171
		−254	483	345	25	16	4	
Copper (C10200, C12200)	SB-75	RT	228	69	45	76	17	251
		−198	359	97	69	102	9	130
90/10 Cu-Ni CDA 706	SB-467	RT	303	110	42		17	45
		−198						
70/30 Cu-Ni CDA 715	SB-467	RT	303	138	40	107	16	29
		−198	586	214	60	118		
Monel 400	SB-165	RT	552	172	42	68	13	26
		−198	793	345	64	68	5	16

* The letters S and H designate soft and hardened conditions respectively.

[1] Corresponding ASTM specifications are B75, B165, B210 and B467.

Source: Praxair, Inc.

Nickel Alloys

While nickel alloys could be used in cryogenic piping systems on the basis of their mechanical properties, their high cost generally precludes their use. One major exception is the use of Monel,® a nickel-copper alloy, which may be used in oxygen systems to minimize ignition tendencies where there is concern of impingement or the potential for high velocities.

NONMETALLIC MATERIALS

Although the bulk of the materials used in cryogenic pipelines are metals, nonmetallic materials have critical functions in cryogenic pipeline components such as valves and insulation. Numerous nonmetallic components have been used in cryogenic pipeline applications, and it would be beyond the scope of this section to cite them all. A brief listing of some of the more common nonmetallic materials used in pipeline components is found in Table C8.10, Table C8.11, and Table C8.11M (Metric).

TABLE C8.10 Typical Examples of Nonmetallic Materials Used in Cryogenic Piping

Application	Material
Gaskets	Durabla (bonded or compressed asbestos) Grafoil (flexible graphite)
Insulation (fiber)	Mineral wool Fiberglass
Insulation (foam)	Polyurethane Styrofoam Foamglass(R)
Insulation (powder)	Perlite Vermiculite
Insulation (sheet)	Mylar Aluminum opacified paper
Insulation (support brock)	Transite Micarta
Valve packing, seals, and lubricants	Viton KEL-F Glass-reinforced Teflon Fluorolube

Source: Praxair Inc.

TABLE C8.11 Typical Mechanical and Physical Properties of Nonmetallic Materials

Material	Tensile strength (ksi)	Modulus of elasticity (ksi)	Specific gravity	Thermal expansion (in/in/°F × 10^{-5})	Thermal conductivity [Btu/(h · ft · °F)]	Specific heat (Btu · lb · °F)
Durabla (asbestos gasket material)	4–11					
Grafoil (flexible graphite)	.75–1.00	200	1.1	0.02	432*	0.7
Mineral wool fiber insulation	0.27		0.15–0.2	0		
Fiberglass fiber insulation	50–500	20,000		0.5–0.8		
Perlite powder insulation						
Vermiculite powder insulation			0.14			
Mylar sheet insulation	17–18 36	700 1600	1.39		0.02	
Aluminum opacified paper sheet insulation	No longer available					
Transite insulation block						

* Through thickness conductivity.
Source: Praxair Inc.

TABLE C8.11M (Metric) Typical Mechanical and Physical Properties of Nonmetallic Materials

Material	Tensile strength (MPa)	Modulus of elasticity (MPa)	Specific gravity	Thermal expansion (m/m/K × 10^{-6})	Thermal conductivity [W/(m · K)]	Specific heat J/(kg · K)
Durabla (asbestos gasket material)	27–76					
Grafoil (flexible graphite)	5–7	1378.	1.1	0.36	747*	2929.
Mineral wool fiber insulation	1.9		0.15–0.2	0		
Fiberglass fiber insulation	344–3440	137,880.		9–14		
Perlite powder insulation						
Vermiculite powder insulation			0.14			
Mylar sheet insulation	117–124 248	4826. 11,030.	1.39		0.035	
Aluminum opacified paper sheet insulation	No longer available					
Transite insulation block	No longer available					

* Through thickness conductivity.
Source: Praxair Inc.

Again, compatibility and mechanical and physical properties must be considered in the selection of nonmetallic materials. With respect to compatibility, liquid oxygen is the commercial cryogen of greatest concern because of its large usage. Typical tests or experimental parameters covered in the selection of nonmetallic materials for oxygen service include *autoignition temperature, heat of combustion, impact test*, and *oxygen index*. Materials that are compatible with oxygen generally have high autoignition temperatures, low heats of combustion, high energy absorption in impact tests, and high oxygen index values. Reference 1 gives advice about the specific criteria levels required for different applications. It is recommended that nonmetallic materials be qualified for oxygen service on a batch-by-batch basis.

Liquid fluorine is of greater concern from the compatibility standpoint with respect to nonmetallic materials. However, the industrial usage of liquid fluorine is very low in comparison to the other industrial gases. Reference 2 should be consulted for information on liquid fluorine systems.

The mechanical and physical properties of significance in the use of nonmetallic materials relate to their application in the form of insulation, gaskets, seals, and lubricants. It may be noted that they are not currently used as structural materials. Development of composites is expected to significantly increase the use of nonmetallic materials as structural components in cryogenic piping.

JOINING

Welding is the most common joining technique used in cryogenic piping. Brazing and soldering may be encountered in cuprous piping. With the recent trends toward high purity, there is a greater tendency to use orbital arc-welded stainless steel piping in lieu of brazed copper alloy piping.

Joint selection, filler metals, process qualification, and welded qualification are covered in detail by the ASME Pressure Piping Code, Section B31.3, Process Piping Code. Generally, areas which have been welded, brazed, or soldered experience thermal effects which may exhibit reduced strength, ductility, or toughness if not properly controlled. Appropriate mechanical tests as specified by applicable codes must be performed to verify suitability.

Within components such as cold boxes there may be occasions when transitions must be made between metals such as stainless steel and aluminum, forming joints which are difficult to weld directly or are unweldable. Transition joints are commercially available which may be classified as mechanical, brazed, diffusion bonded, or explosion bonded. Such joints have been successfully used in cryogenic applications. However, caution is advised in order to avoid in-service problems such as leaks, embrittlement, or actual joint separation. Careful vendor evaluation, joint design evaluation, and attention to fabrication details are required when dissimilar metal joints are required.

ENVIRONMENTAL AND SAFETY CONCERNS

A number of metallic and nonmetallic materials may be toxic, carcinogenic, tetragenic, or have other properties which are considered environmentally undesirable (i.e., effects on the ozone layer). For example, the use of asbestos-containing materials such as Durabla or certain vermiculite ores may be further restricted or phased out. Similar concerns exist with cadmium-bearing brazing alloys. The recent concern about ozone depletion caused by chlorofluorocarbons could affect polyurethane foam production. Consequently, many common materials currently used in cryogenic piping systems are likely to be phased out over time.

Recent legislation mandates that material safety data sheets (MSDS) be obtained for all industrial substances that are in industrial use. This obviously includes the metallic and nonmetallic materials used in cryogenic piping. Failure to comply with the requirements and implementation provisions of the original legislation can have serious consequences for fabricators.

Equipment used in oxygen services has additional requirements such as cleaning and velocity limitations which must be considered. Oxygen equipment must be cleaned to eliminate contaminants such as hydrocarbons and metal particulates, which could serve as ignition sources. Reference 3 provides a general discussion of this issue. In addition, velocity limitations may be placed on certain classes of material to preclude ignition by particle impingement. See Reference 4 for velocity limitations and design considerations pertinent to oxygen systems.

Other industrial gases that might be encountered in cryogenic pipelines have specific hazards associated with them (i.e., flammability, toxicity, etc.). These are summarized in MSDS forms available from industrial gas suppliers.

REFERENCES

1. ‘‘Guide for Evaluating Nonmetallic Materials for Oxygen Service,’’ ASTM Specification G63, American Society for Testing and Materials, Philadelphia, PA.
2. ‘‘Design Handbook for Liquid Fluorine Ground Handling Equipment,’’ Technical Report No. AFRPL-TR-65–133, Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, 1965.
3. J.G. Hust and Cark, A.F., ‘‘A Survey of Compatibility of Materials, with High Pressure Oxygen Service,’’ *Cryogenics*, vol. 13, 1973, pp. 325–335.
4. ‘‘Cleaning Equipment for Oxygen Service, ’’ CGA Pamphlet G.4.1, Compressed Gas Association, Arlington, VA.

BIBLIOGRAPHY

Handbook on Materials for Superconducting Machinery, Technical Report MCIC-HB-04, Metal & Ceramics Information Center, Battelle, Columbus, OH, November 1974.

Metals Handbook (desk edition), American Society for Metals, Cleveland, OH, 1985, pp. 20.24–20.34.

Reed, R. P., and Clark, A. F., ‘‘Materials at Low Temperatures,’’ American Society for Metals, Metals Park, OH, 1983.

Schwartzberg, F. R., *Cryogenic Materials Data Handbook*, vols. I and II, Technical Report AFML-TR-64–280, Wright Patterson Air Force Base, OH, July 1970.

PIPING SYSTEM DESIGN—FLUIDS

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PIPE SIZING CRITERIA

For all cryogenic fluids, except oxygen gas, pipe sizing is based on pressure drop considerations (for oxygen gas, see the section "Oxygen Gas Piping"). The pressure drop criteria presented in Chap. B8 are applicable to cryogenic fluids when the fluid is either in the liquid phase or gas phase flow regime.

Heat transfer into a saturated cryogenic liquid or a drop in pressure can cause a portion of the liquid to flash into a gas (see the section "Properties of Cryogenic Fluids"). A two-phase (gas-liquid) flow condition must then be considered when determining the required pipe size and pressure head requirements (see the next section). Two-phase flow will result in larger pressure drop losses or a larger pipe size requirement than will liquid phase flow. Therefore, whenever possible, the fluid should be maintained in a single-phase flow condition. The liquid can be subcooled to prevent flashoff due to pressure decreases such as across throttling valves. Use of pipe insulation can reduce the heat leak into the piping, thereby also reducing flashoff.

TWO-PHASE FLOW

For prediction of the pressure drop experienced by a two-phase cryogenic fluid flowing along a pipe, the usual practice is to divide the pipe into sections for calculation purposes (see Fig. C8.4). At the beginning of a given pipe section,

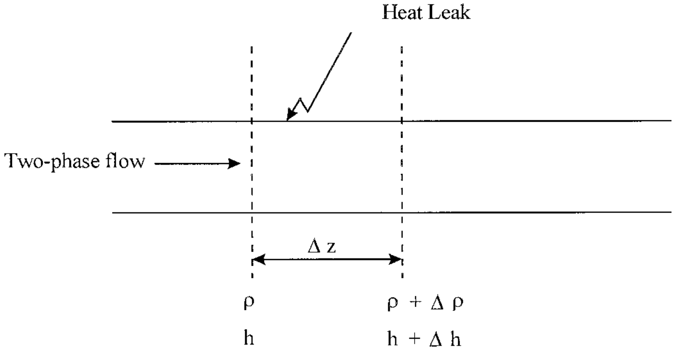

FIGURE C8.4 Pipe section.

conditions are assumed to be known. The pressure drop over the section is then calculated based on the conditions at the beginning of the section, and so the pressure at the end of the section can be determined. The heat leak into the section is estimated from heat transfer correlations. An enthalpy balance over the section then allows the enthalpy (h) at the end of the section to be calculated (potential and kinetic energy changes over the section can usually be neglected). Knowledge of the pressure and enthalpy at the end of the section allows new conditions to be calculated from the thermodynamic properties of the cryogenic fluid. Such conditions include the temperature, vapor fraction, and physical properties of each phase. The calculations are then repeated for the next section, and so on to the end of the pipe.

Two-Phase Pressure Drop

The total pressure drop for two-phase flow consists of three parts; the frictional, gravitational, and accelerational pressure drops, so that,

$$\left(\frac{dp}{dz}\right)_{TP} = \left(\frac{dp_F}{dz}\right) + \left(\frac{dp_G}{dz}\right) + \left(\frac{dp_A}{dz}\right) \tag{C8.1}$$

where p is the pressure, $p$dl ft^{-2} (Nm^{-2})and z is the distance along the pipe, ft (m).

Many correlations have been proposed to predict the frictional pressure drop (dp_F/dz) and they are discussed in Chap. B8. The earliest correlation, which is still widely used, is that of Lockhart and Martinelli.[1]

To use this correlation, the steps are:

1. Calculate the Reynolds number for each phase flowing alone in the pipe.

$$\mathrm{Re}_L = \frac{M_L D}{A\mu_L} \tag{C8.2}$$

$$\mathrm{Re}_G = \frac{M_G D}{A\mu_G} \tag{C8.3}$$

where M_L, M_G = liquid and vapor flow rates, lbs^{-1} (kg s^{-1})
A, D = pipe cross sectional area, ft^2 (m^2), and diameter, ft (m)
μ_L, μ_G = liquid and vapor viscosities, lb ft^{-1} s^{-1} (Nsm^{-2})

2. Calculate the frictional pressure drop for each phase flowing alone in the pipe.

$$\left(\frac{dp_F}{dz}\right)_L = \frac{2k_L(\mathrm{Re}_L)^{-n}\rho_L}{D}\left(\frac{M_L}{A\rho_L}\right)^2 \tag{C8.4}$$

$$\left(\frac{dp_F}{dz}\right)_G = \frac{2k_G(\mathrm{Re}_G)^{-m}\rho_G}{D}\left(\frac{M_G}{A\rho_G}\right)^2 \tag{C8.5}$$

Where ρ_L, ρ_G = liquid and vapor densities, lb ft^{-3} (kg m^{-3}).

The following table can be used to determine k_L, k_G, n, and m:

Liquid	Vapor	Re_L	Re_G	k_L	k_G	n	m
t	t	>2,000	>2,000	0.046	0.046	0.2	0.2
v	t	<1,000	>2,000	16	0.046	1.0	0.2
t	v	>2,000	<1,000	0.046	16	0.2	1.0
v	v	<1,000	<1,000	16	16	1.0	1.0

The value of 0.046 is based on smooth pipes, and *t* and v denote turbulent or laminar flow respectively.

3. Calculate X from

$$X = \left[\left(\frac{dp_F}{dz}\right)_L \bigg/ \left(\frac{dp_F}{dz}\right)_G\right]^{0.5} \tag{C8.6}$$

4. Use Fig. C8.5 to determine ϕ_L or ϕ_G.

5. Calculate the frictional pressure drop for two-phase flow from either,

$$\left(\frac{dp_F}{dz}\right) = \phi_L^2\left(\frac{dp_F}{dz}\right)_L \quad \text{or} \quad \left(\frac{dp_F}{dz}\right) = \phi_G^2\left(\frac{dp_F}{dz}\right)_G \tag{C8.7}$$

This correlation typically allows prediction of the frictional pressure drop to about ±30%, but the discrepancy can be greater under some circumstances.

Where the flow is other than horizontal, the gravitational pressure drop becomes significant, and in vertical flow it is the dominant term. It may be calculated from

$$\left(\frac{dp_G}{dz}\right) = g \sin\theta[\alpha\rho_G + (1-\alpha)\rho_L] \tag{C8.8}$$

where g = acceleration due to gravity, ft s^{-2} (ms^{-2})
α = void fraction
θ = angle to horizontal, deg.

Numerous correlations are available for the void fraction, and that of Lockhart and Martinelli is shown in Fig. C8.5. As it was derived originally for horizontal flow, it can be rather inaccurate for vertical flow. Other void fraction correlations are mentioned in Chap. B.8.

The accelerational pressure drop is often negligible for two-phase flow of cryogenic fluids. It can be estimated from,

$$\left(\frac{dp_A}{dz}\right) = \frac{1}{A^2}\frac{d}{dz}\left[\frac{M_G^2}{\rho_G\alpha} + \frac{M_L^2}{\rho_L(1-\alpha)}\right] \tag{C8.9}$$

where the expression on the right-hand side is evaluated over the pipe section.

The total pressure drop calculated by this method should be increased by at

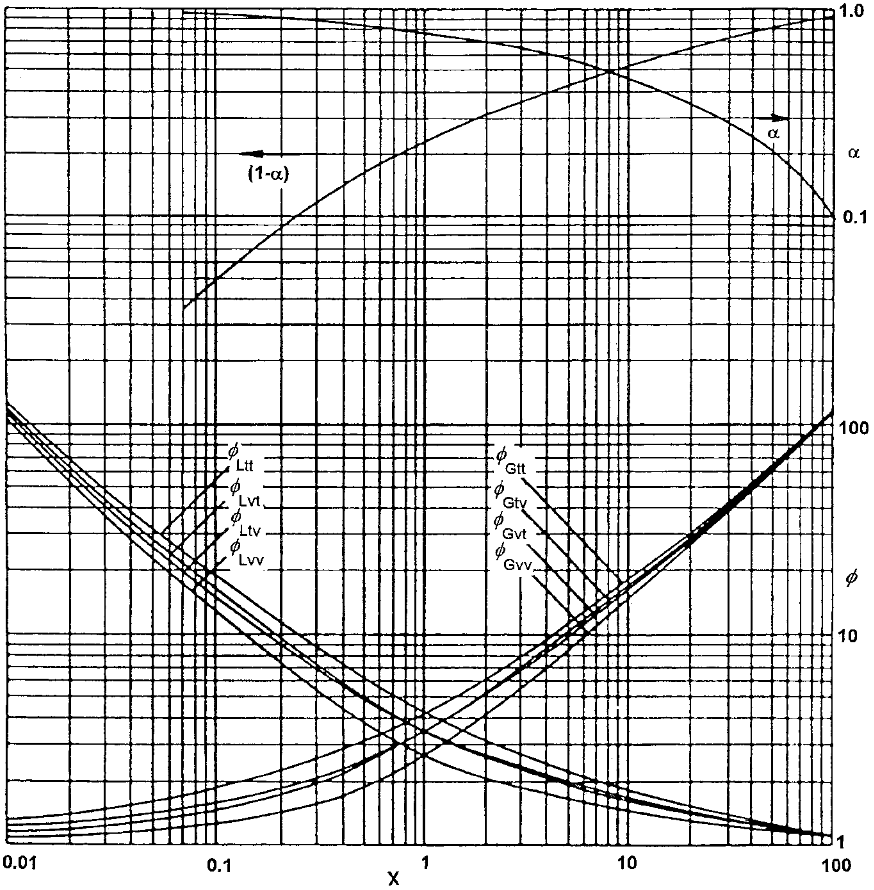

FIGURE C8.5 Lockhart-Martinelli correlation.

least 30% for design safety. A final check should also be made for critical, or choked, flow at the downstream end of the pipe. An equation proposed by Schweppe and Foust[2] for the maximum possible flow rate is

$$M_c = -\left[\frac{A^2}{\left(\frac{\partial v}{\partial p}\right)_s}\right]^{0.5} \tag{C8.10}$$

where M_c = maximum flow rate of the mixture, lbs^{-1} (kg s^{-1}).

v = specific volume of the mixture based on homogenous flow, $\text{ft}^3\ \text{lb}^{-1}$ ($\text{m}^3\ \text{kg}^{-1}$).

so that

$$v = \frac{M_G / \rho_G + M_L / \rho_L}{M_L + M_G} \tag{C8.11}$$

and the partial derivative is evaluated under isentropic conditions. It can be calculated for conditions at the downstream end of the pipe from the thermodynamic properties of the cryogenic fluid.

An alternative version, involving the enthalpy, h, which is sometimes more convenient, is

$$M_c = -\left[\frac{A^2}{\left(\frac{\partial v}{\partial p}\right)_h + v\left(\frac{\partial v}{\partial h}\right)_p}\right]^{0.5} \tag{C8.12}$$

where the two partial derivatives are evaluated at constant enthalpy and constant pressure respectively.

If M_c is less than the assumed total flow rate, the pressure drop calculation should be repeated with a reduced total flow rate or an increased pipe diameter.

OXYGEN GAS PIPING

For oxygen gas, the fluid velocity must also be considered when determining the pipe size. The allowable velocity is a function of the oxygen gas pressure and temperature and the material of the piping and its components. For systems with pressures up to 1000 psi (69 bar) and a maximum temperature of 200°F (366 K), carbon steel and stainless steel piping are acceptable provided that the maximum allowable gas velocity as shown in Fig. C8.6 is not exceeded.

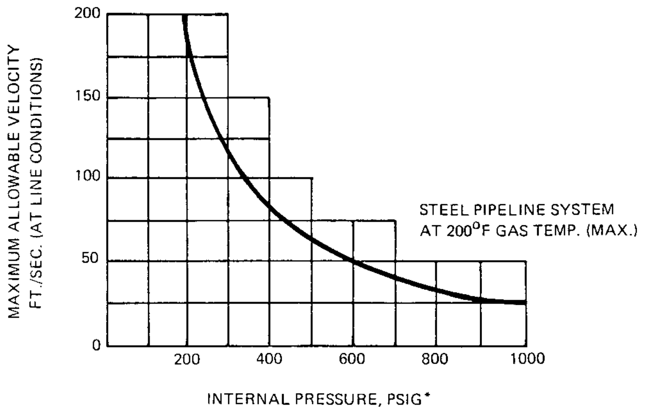

FIGURE C8.6 Maximum velocity versus internal pressure for steel pipelines. (Ref. 3.)

When the velocity is exceeded, copper or a copper-base material such as brass or monel is required. The velocity criteria should also be considered at valves. For example, sonic velocity can occur at throttling and safety valves. Copper-base materials are required for the parts of the valves where high velocity can occur.

When oxygen gas impinges directly on ferrous piping, such as from a side back feed line, the allowable velocity must be reduced to one-half the values given in Fig. C8.6, or the impingement surface must be a copper-base alloy. When the velocity returns to acceptable levels, the copper base alloy is extended for 8 diameters in pipe length before returning to ferrous piping. For a more thorough discussion of material requirements for oxygen service, see Ref. 3 at the end of this section.

PIPING ARRANGEMENT

Consideration of the cryogenic fluid properties has an effect on the piping arrangement. Because the cryogenic fluid is colder than ambient air, the continuous heat leak from ambient air to the piping system is a design consideration.

Whenever cryogenic liquid can be trapped between two valves, a line block safety valve must be provided to prevent overpressurization caused by heat transfer from the ambient air to the cryogenic fluid. As noted earlier in the discussion of cryogenic fluids, containment of the pressure increase caused by vaporization of the trapped cryogenic liquid is not practical and use of a line block safety valve is mandatory. When cryogenic gas can be trapped between two valves, the pressure rise should be calculated to ensure that the piping system design pressure is not exceeded when the trapped gas warms up to ambient temperature. When the design pressure can be exceeded, a line block safety valve should be used.

Traps are normally designed into liquid piping systems to prevent undesirable heat leak from a branch of the piping system when it is not in use. To form a trap at the branch and main run connection, a vertical rise equal to a minimum of 2 pipe diameters is provided in the branch. Heat transfer into the nonflowing branch will cause the liquid to vaporize and a gas-liquid interface will form in the trap. The gas-liquid interface in the vertical rise will prevent flow from the main run into the nonflowing branch. Undesirable heat transfer and liquid loss is therefore reduced. When liquid flow is required through the branch, the liquid will flow through the trap.

When a cryogenic liquid line is initially put in service, the warm piping will cause *liquid flashoff,* which could restrict the flow during the two-phase flow transient period. When it is possible to precool the lines, the piping can be sized for liquid phase flow, which will result in smaller piping. If rapid cooldown is required, the piping must be sized for two-phase flow.

As a good approximation, the amount of liquid required to cool down a line is

$$W = \frac{M_m C_{pm} \Delta T_m}{(H_v - H_l)} \tag{C8.13}$$

where W = liquid, lb (kg)
M_m = line to be cooled, lb (kg)
C_{pm} = mean specific heat of line, Btu/(lb · °F), (kJ kg^{-1}K^{-1})
ΔT_m = temperature change through which line is cooled, °F(K)
H_v = enthalpy of fluid as a vapor at ambient conditions, Btu/lb (kJ kg^{-1})
H_l = initial enthalpy of liquid when entering line, Btu/lb (kJ kg^{-1})

REFERENCES

1. R.W. Lockhart and R.C. Martinelli, *Chem. Eng. Prog.,* ***45,*** 39, 1949.
2. J.L. Schweppe and A.S. Foust, *Chem. Eng. Prog.,* ***49,*** Symp. Ser. 5, 77, 1953.
3. CGA Pamphlet G-4.4, "Industrial Practices for Gaseous Oxygen Transmission and Distribution Piping Systems," Compressed Gas Association, Inc. Arlington, VA.

PIPING SYSTEM DESIGN—MECHANICAL

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APPLICABLE CODES AND MATERIALS

The applicable code for the design of cryogenic piping systems is the ASME B31.3 Process Piping Code.[1] B31.3 presents a design procedure to determine the stresses in the pipe due to fluid pressure, pipe and fluid weight, and thermal expansion and contraction of the pipe. The code also lists piping materials by ASTM specification number. The allowable stress as a function of design temperature is listed for each material. The listed minimum design temperature is used to determine which materials are suitable for cryogenic service.

Some of the materials can be used in lower-temperature service, provided the specified impact test is performed to ensure adequate ductility at the lower temperature. The B31.3 Code also lists pipe fittings and bolting by ASTM number so that materials compatible with the pipe can be selected. For more discussion of materials, see the section "Materials Used in Cryogenic Piping Systems."

Other piping design considerations such as corrosion resistance, chemical compatibility, and material melting point can affect the material selection. Some examples of these conditions include:

1. Copper-base materials are not recommended for ammonia or acetylene service because copper alloys react with the fluid.
2. Stainless steel piping is recommended for flammable fluids such as hydrogen rather than lower-melting-point materials such as copper or aluminum unless the piping is protected to prevent exposure to heat in the event of a fire.
3. Copper-base materials (such as monel) are recommended for high-velocity oxygen service.

See Chap. B2 for discussion of formulas which can be used to calculate the pipe wall thickness as required to contain the fluid pressure.

ECONOMIC PIPE SIZING

When a piping system is being designed, an initial pipe size must be selected before the piping configuration is developed. This subsection provides recommendations for selecting the initial pipe size. However, after the piping system is developed, the pipe size selection is finalized by calculating the pressure drop based on the actual piping configuration and comparing it to the pressure head available.

For cryogenic liquid lines, the initial pipe size should be selected so that the liquid velocity is in the range of 5 ft/s (1.5 m/s).

For cryogenic liquid lines, the initial pipe size should be selected based on the available pressure head (see section "Piping System Design—Fluids," subsection "Pipe Sizing Criteria"). When estimating pipe length and number of fittings to calculate the initial pipe size, add 50 percent to the total resistance coefficient, *K*, to allow for pipe loops and extra fittings as may be required for thermal flexibility. To ensure good process control, one-third of the total system pressure drop should be allotted to the control valves.

Except for oxygen gas, as discussed in the section "Piping System Design—Fluids," subsection "Oxygen Gas Piping," there are no specific gas velocity limitations. However, the gas velocity is normally less than 400 ft/s (130 m/s) to avoid noise problems and excessive pressure drop. For systems with pressures up to 400 psi (2750 KPa), gas velocities in the range of 50 to 200 ft/s (15 to 65 m/s) are common.

When the pipe size of the gas line affects the plant power consumption, the initial pipe size can be selected by using the following equation. The equation calculates the pipe diameter that provides the minimum total cost; that is, pipe capital cost plus operating power cost.

Economical pipe sizing (Imperial units)

$$D = (0.0275)\left(\frac{MTUCY}{EA}\right)^{0.16}\frac{Q^{0.48}}{P^{0.32}} \qquad \text{(C8.14)}$$

where M = molecular weight
T = absolute temperature, K
U = average compression temp., K
C = cost of power, \$/kW · h
Y = evaluation period, years
E = compressor overall efficiency
A = pipe cost fraction, \$ per 100-ft/in pipe diameter
Q = flow rate, ft^3/h @ 70°F, 14.7 psia
P = absolute pressure, psia
D = inside pipe diameter, in

Economical pipe sizing (Metric units)

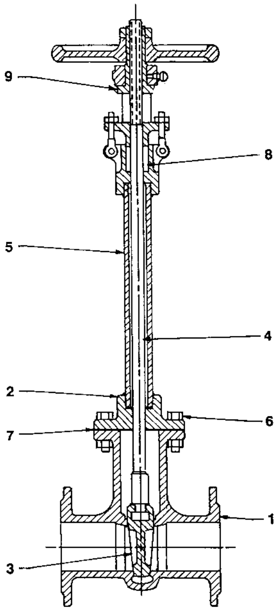
$$D = (7.254)\left(\frac{MTUCY}{EA}\right)^{0.16}\frac{Q^{0.48}}{P^{0.32}} \qquad \text{(C8.14M)}$$

where M = molecular weight
T = absolute temperature, K
U = average compression temp., K
C = cost of power, \$/megajoule
Y = evaluation period, years
E = compressor overall efficiency
A = pipe cost fraction, \$ per 100-m/mm pipe diameter
Q = flow rate, m^3/h @ 0°C
P = absolute pressure, KPa
D = inside pipe diameter, mm

PIPING COMPONENTS

The types of valves used in cryogenic service are similar to those used for conventional fluids, except that the valves may require unique design features due to the cryogenic fluids. Metallic and nonmetallic materials must be suitable for the low-temperature service. See the discussions on materials in the section ''Materials Used in Cryogenic Service.'' Valve types used include gate, globe, butterfly, ball, check, and safety.

The stems of gate, globe, butterfly, and ball valves are extended so that the valve packing and operator remain at ambient temperature when cryogenic fluid is in the valve body. The valves are oriented so that the packing is at a higher elevation than the valve body, so that a gas pocket can form inside the valve's extension tube, thereby making the extension tube more effective at insulating the packing from the cold temperatures. Figure C8.7 shows an extended stem gate valve.



LEGEND

ITEM NO.	PART NAME	ITEM NO.	PART NAME
1	BODY	6	BONNET BOLTING
2	BONNET	7	BONNET GASKET
3	WEDGE	8	PACKING
4	STEM	9	YOKE BUSHING
5	EXTENSION TUBE		

FIGURE C8.7 Extended stem gate valve. *(Courtesy Praxair, Inc.)*

Valves must be designed so that liquid cannot be trapped in a portion of it when the valves are cycled from open to closed. When a conventional gate valve is closed, it is possible for liquid to be trapped in the bonnet area. If liquid is trapped, heat input from the atmosphere will cause the liquid to vaporize and overpressurize the valve body. When a gate valve is used in cryogenic service, the bonnet area is vented to one side of the valve so that any vaporized gas cannot be trapped in the bonnet area. Figure C8.8 shows one method that is used to vent the bonnet area of a gate valve.

With a conventional ball valve, liquid can be trapped between the ball and the

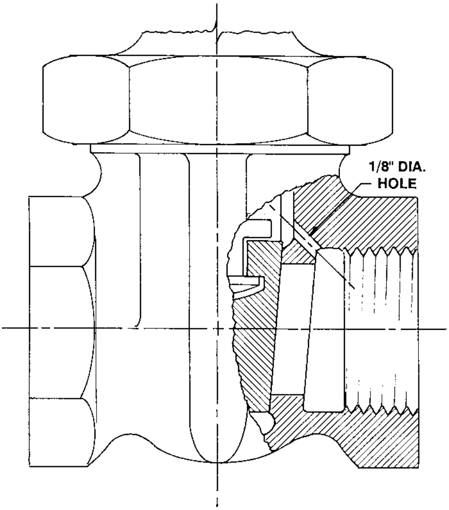


FIGURE C8.8 Gate valve—vented bonnet. *(Courtesy Praxair, Inc.)*

seat. Similarly, for ball valves in cryogenic service, the ball area is vented to one side of the valve.

During the cool-down or thaw of a cryogenic piping system, different parts of the valves may cool down or warm up at different rates, resulting in varying rates of contraction and expansion. The valve design must consider that the valves are required to be operational during these conditions.

FLEXIBILITY ANALYSIS AND SUPPORTS FOR CYROGENIC PIPING SYSTEMS

Piping flexibility analysis is an important design consideration because the large difference between ambient and cryogenic temperatures will result in significant thermal contraction. The analysis methods used are similar to those required for conventional piping systems as discussed in Chap. B4. The one difference is that piping in cryogenic service contracts rather than expands as is the case with high-temperature service. However, since the commercially available flexibility analysis computer programs have the temperature range as an input value, and the program calculates the resulting thermal contraction, the analysis methods become identical to those used for conventional piping systems.

When possible, cryogenic piping is routed so that the piping configuration provides adequate flexibility. Generally, piping is routed in a manner that includes Z, L, and U bend configurations to take up the pipe movement while keeping the stresses within the allowable range. When additional flexibility is required, the flexibility can be increased by the addition of expansion loops (U bends) or the addition of flexible metal hose. Flexible hose can accommodate larger pipe movements and can result in a more compact design. However, the use of flexible hoses may result in requiring more pipe supports to guide the piping so that the pipe movement is taken up by the hose.

The flexible hose is located so that pipe movement is lateral to it. The braided cover on the flexible hose prevents any significant pipe movement axial to the flexible hose. A flexible hose acts similarly to an expansion joint designed for lateral movements. The recommended upper pressure limit for flexible hose and expansion joints is 1000 psi (6900 kPa).

Hanging-style pipe supports can be used to accommodate significant pipe movement in both the lateral and axial directions. Roller-style pipe supports can be used to accommodate large axial pipe movement. When the amount of pipe movement exceeds the capability of a hanger or roller pipe support system, a fixed support located in the center of the pipe span can be effective in reducing the amount of movement.

When an uninsulated cryogenic line is supported, a portion of the pipe support will be at cryogenic temperature. The lower temperature should be considered when selecting the materials for the pipe support and its hardware.

INSTALLATION OF PIPING COMPONENTS

To minimize potential leaks, welding and brazing are the most common assembly methods for cryogenic piping systems. In accordance with the B31.3 Code, solder joints are not acceptable for cryogenic service. However, to allow removal of valves and other piping components for maintenance, other assembly methods are used at piping components.

Piping components with threaded fittings are used in small sizes, usually NPS 1½ pipe size and smaller. Flange joints are used for large sizes, with flanges provided in accordance with ASME B16.5. For raised-face and flat-face flanges, gaskets are usually compressed asbestos sheets, spiral-wound stainless steel with asbestos or Teflon filler, or flexible graphite such as Grafoil.

INSULATION SYSTEMS

Most piping in liquid cryogenic service is insulated. The only reasons a line would not be insulated are that (1) its use is very infrequent and brief; (2) it is a temporary installation; or (3) the refrigeration losses are inconsequential. The following table shows the heat leak from a frosted, uninsulated line containing liquid nitrogen and subjected to an 8 mi/h (3.6 m/sec) wind:

Pipe size (in)	Heat leak per foot of pipe (BTU/hr)	Pipe size (mm)	Heat leak per meter of pipe (Watt)
1	280	25	270
2	530	50	510
4	1000	100	960

The type of insulation used for cryogenic piping includes (1) expanded foams such as polyurethane and foamglass, (2) powder insulations such as perlite, and (3) vacuum-insulated pipe. For an insulation system to remain effective, the vapor barrier system must keep atmospheric moisture from entering the insulation space and freezing against the cryogenic line. When this occurs, the ice that is formed will degrade or destroy the insulation system.

When the cryogenic liquid is colder than the boiling point of oxygen (−297°F or −183°C), oxygen can condense out of the air and collect in the insulation space. For this situation, the insulation system should be noncombustible in the presence of oxygen.

TABLE C8.12 Thermal Conductivity of Pipe Insulation Materials at an Insulation Mean Temperature of −100°F

Insulation	Thermal conductivity [Btu/(h · ft · ² °F)]	Thermal conductivity W/(m · °K)
Urethane foam	0.012	.021
Foamglass	0.024	.042
Perlite (at atmospheric pressure)	0.018	.031
Perlite (vacuum at 1 μm)	7.9×10^{-4}	1.37×10^{-3}
Laminar radiation shielding (vacuum at 0.1 μm)	2.1×10^{-5}	3.63×10^{-5}

The typical values for thermal conductivity are shown in Table C8.12. The expanded foam insulations use a plastic covering, such as PVC or neoprene sheeting, to provide the vapor barrier protection. The initial capital cost is usually lower than the other systems, but more frequent maintenance is required to maintain a tight vapor barrier.

Powder insulation is generally used when several piping segments and pieces of equipment can be grouped in one area. A metal jacket or casing is used to contain the perlite around the piping and equipment. When the insulation space is maintained at atmospheric pressure, it must be purged with a dry gas, such as nitrogen, to keep atmospheric moisture out of the casing. For improved heat transfer performance, the casing can be made vacuum-tight and the insulation space evacuated to a high vacuum. For powder insulation, high vacuum is a pressure level less than 1 μm of mercury when the line is at the normal cryogenic temperature operation condition (1 μm of mercury is equal to 10^{-3} torr). See Table C8.12 for typical thermal conductivity values. When considering this insulation option, the difficulty in maintaining a vacuum-tight casing must be weighed against the difficulty of maintaining a dry gas purge, considering the difference in heat transfer performance.

Vacuum-insulated piping (VIP) is constructed of a stainless steel inner pipe that contains the cryogenic fluid, a stainless steel outer jacket to form the vacuum space, and insulation in the vacuum space. The insulation is normally laminar radiation shielding that consists of alternate layers of a reflective material, such as aluminum foil, and an insulation material, such as glass paper. For vacuum-insulated pipe, the required vacuum is a pressure level less than 0.1 μm, when the line is at the normal cryogenic operating condition. Heat leak by conduction and radiation is reduced by the laminar radiation shielding. The heat leak by convection is reduced by the vacuum.

Nonmetallic spacers are required in the vacuum space to support and maintain alignment of the inner pipe within the outer pipe jacket. Bellows are required in one pipe to account for the differential expansion between the inner and outer

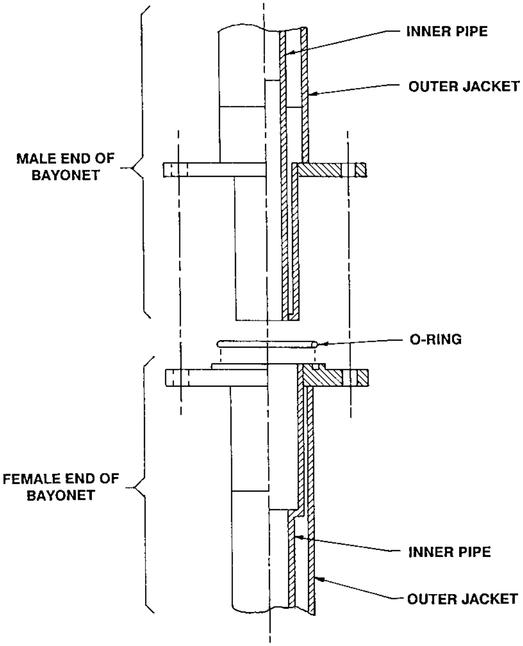


FIGURE C8.9 Vacuum-insulated pipe bayonet assembly. *(Courtesy Praxair, Inc.)*

TABLE C8.13 Typical Heat Leak Values for Vacuum-Insulated Pipe when the Fluid Temperature is −320°F (−196°C)

Pipe size (in)†	Pipe	Elbow	Tee	Flex hose	Bayonet and field-welded joints	Valve
¾	0.47	1.50	2.00	1.41	15	14
1	0.50	2.30	3.40	1.50	21	15
1½	0.58	2.60	4.00	1.74	23	19
2	0.64	3.00	4.50	1.92	24	25
3	0.79	3.80	5.70	2.37	48	64
4	0.92	4.40	6.50	2.76	84	79
6	1.20	6.00	9.00	3.60	110	120
8	1.46	7.40	11.00	4.38	140	180

* (Heat leak values are BTU/h/ft of pipe and flex hose and are BTU/h for each fitting and valve.)

† For the inner pipe.

TABLE C8.13M (Metric) Typical Heat Leak Values for Vacuum-Insulated Pipe when the Fluid Temperature is −320°F (−196°C)*

Pipe Size (mm)†	Pipe	Elbow	Tee	Flex hose	Bayonet and field-welded joints	Valve
19	0.45	1.44	1.92	1.36	4	4
25	0.48	2.21	3.27	1.44	6	4
38	0.56	2.50	3.85	1.67	7	6
50	0.62	2.88	4.33	1.85	7	7
75	0.76	3.65	5.48	2.28	14	19
100	0.88	4.23	6.25	2.65	25	23
150	1.15	5.77	8.65	3.46	32	35
200	1.40	7.12	10.60	4.21	41	53

* (Heat leak values are watts/m of pipe and flex hose and are watts for each fitting and valve.)
† For the inner pipe.

pipe. Due to the labor-intensive nature of fabricating VIP, it is normally shop-fabricated. To accommodate field installation, a mechanical joint is required between pipe segments. A bayonet assembly is one type of joint that has an extended heat leak path between the inner pipe and the flange connection of the outer pipe. Figure C8.9 illustrates the bayonet assembly.

Table C8.12 shows typical values of insulation thermal performance for laminar radiation shielding. For a vacuum-insulated piping system, the thermal performance of the straight pipe is affected by the number and type of spacers. The thermal performance of fittings and other components depend on fabrication techniques. Tables C8.13 and Table C8.13M (Metric) show typical heat leak performance values for commercially available vacuum insulated pipe.

The total cost, including the piping system and the cost of refrigeration losses due to heat leak, should be considered when selecting an insulation system. For many permanent piping systems, vacuum-insulated pipe provides the most cost-effective insulation system.

CLEANING

All materials used in oxygen piping systems or connected with oxygen systems should be cleaned before the system is put into service. The cleaning must remove mill scale, rust, dirt, weld slag, oil, grease, and other organic materials. The purpose of the cleaning is to remove hazardous hydrocarbons and particulate contaminants which could ignite and cause a fire in an oxygen atmosphere. To obtain additional information on cleaning requirements, see Ref. 2. For a discussion of the factors affecting ignition in an oxygen system, see Ref. 3.

All materials used in nonoxygen cryogenic systems are also cleaned before the system is put in service. The purpose of the cleaning is to reduce contaminants to the point where they will not migrate, seize up moving parts at low temperatures, or prevent the attainment of high product purity or vacuum levels.

VENTS

When a cryogenic fluid is vented, such as from safety valves, safe disposal of the fluid must be considered. The fluid should be directed so that it will not contact personnel, because the low temperature can cause burns.

When cold, most cryogenic fluids are heavier than air. A heavier-than-air gas can displace the air and create a potential for asphyxiation. Some cryogenic fluids, such as hydrogen, are flammable. An oxygen-enriched atmosphere can promote flammability of other materials. Therefore, all vents should be located outdoors and directed so that high concentrations will not collect in confined areas.

REFERENCES

1. ASME B31.3, Process Piping, ASME Code for Pressure Piping, B31 An American National Standard, 1996 edition.
2. CGA Pamphlet G-4.1, *Cleaning Equipment for Oxygen Service,* Compressed Gas Association, Arlington, VA.
3. ASTM G88, Standard Guide for Designing Systems for Oxygen Service, American Society for Testing and Materials, Philadelphia, PA 1984.

COLD BOX PIPING

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A *cold box* is a mechanical system that insulates an entire low-temperature process mechanical embodiment in a single assembly instead of insulating each pipe and vessel individually. The cold equipment and piping are installed in an airtight steel insulation casing. The void space inside the casing is filled with insulation powder or fibers.

The design of cold box piping requires knowledge of some basic engineering disciplines, such as strength of materials, fluid mechanics, and heat transfer. Also required are some piping design skills such as flexibility analysis, pressure drop calculations, and material selection. These skills are discussed in Chap. B4 of this handbook.

For safe design, cold box piping is usually designed to meet the engineering requirements of ASME B31.3[1], Process Piping Code. Further, the designer should be aware of the design considerations for safe operation and maintenance of cold boxes discussed in Sec. 8 of Ref. 2 of this article.

What follows is a description of problems unique to the design of cold box piping, and, where appropriate, suggested methods of solution.

DESIGN REQUIREMENTS FOR COLD BOX PIPING

General System Architecture

Cold boxes tend to be tall, vertical structures. They often contain tall distillation columns and brazed aluminum heat exchangers which are oriented for vertical flow (see Fig. C8.10). A minimum of 12 in (30 cm) of space is usually provided between the casing and the piping and equipment inside. This space provides adequate insulation for the cold equipment and access to the piping for fabrication, testing, and maintenance. Valves and controls above grade level are located so as to be easily accessible from operating platforms. The piping arrangement within the cold box is very compact to minimize the amount of insulation and to fit shop-fabricated cold boxes within the maximum outline which can be shipped by rail or by truck. Heat exchangers operating in parallel are located at the same elevation to avoid flow unbalance due to density differences between the warm and cold streams.

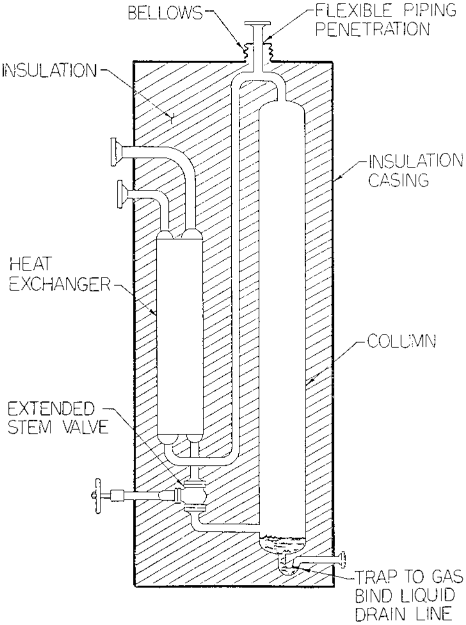

FIGURE C8.10 Schematic cold box diagram. *(Courtesy Praxair, Inc.)*

Insulation System

Before start-up, the insulation is purged with a dry inert gas, usually nitrogen. A small flow of purge gas is continued during operation to maintain a slight positive pressure in the casing and to prevent inflow of oxygen and moisture. Pipe and valve stem penetrations through the casing must be airtight.

Equipment and pipes operating at different temperatures must be separated to minimize unwanted heat flows. The piping and pipe supports must withstand loads from the insulation material. These loads occur when fibrous insulation is being packed into the casing and when powder insulation is being loaded or removed.

Reliability

Piping inside the cold box is not readily accessible for maintenance. Repair of a simple leak requires that the cold box be out of service for several days. Erosion caused by insulation and powder propelled by the leaking fluid can enlarge a small leak or cause another leak in nearby equipment. For these reasons, leak-tight integrity of cold box piping is extremely important. The number of flanged joints inside a cold box is kept to a minimum. Welded or brazed pipe joints are used wherever possible. Transition joints are often used to join stainless valves to aluminum pipe. Weld-end, top-entry, extended-stem globe valves afford maximum reliability. These valves do not have any flanges inside the casing, and the plug and seat can be replaced from outside the cold box. When flanges are used inside the casing, proper torquing of flange bolts must be assured. Threaded joints and tubing compression fittings are usually not used inside the casing. Flanges are sometimes installed in a separate, small, insulated compartment where access can be achieved without removing the insulation from the entire cold box. A careful leak test in done on all piping inside the casing before the insulation is installed.

Thermal Expansion and Contraction

Typically, cold box piping is heated to 212°F (100°C) before start-up to remove any moisture, and then cooled to operating temperature—as low as −320°F (−195°C), as is the case in an air separation plant. In that example, the total temperature range is 532°F (295°C). The total thermal expansion and contraction of aluminum pipe in this range is 6 in/100 ft (0.5 cm/m). The flexibility analysis of the piping must consider the full temperature range as well as any more severe differential temperature conditions which may occur during upset, thaw, or cool-down. Clearance must be allowed for the pipes to move through their full expansion and contraction range. This clearance is especially critical for pipes connected near the top of tall vessels. Pipe penetrations through the casing will restrict the movement of the pipe unless the casing is made flexible by use of a metal bellows or a rubber boot.

Piping Installation Details

Pipes running from liquid pipes or vessels to the casing must have thermal traps (upward rise of at least 2 pipe diameters) to gas-bind the connection and keep the cold liquid away from the warm casing. Thermal traps are also used in gas pipes to prevent unwanted convection currents.

Extended stem valves used in cold pipes are installed with the body located at least 12 in (30 cm) inside the casing and with the packing gland and the operator or handwheel located outside. Liquid valve stems are tilted upward at least 15° to gas-bind the stem extension tube and keep the liquid away from the packing.

Supports for cold pipes are commonly made from austinitic stainless steel because it is strong, ductile at cryogenic temperature, and has a relatively low thermal conductivity. Blocks of insulation material can be included in the supports to reduce heat leak from the casing.

Low-point drains are provided where necessary to remove process liquids for shutdown and to remove liquid water formed during thaw of the cold box.

Cleaning

Cold box piping is cleaned to remove contaminants (see the earlier discussion of cleaning in this chapter). The cleaning is usually done before the pipe is installed. Pipe ends and other openings are covered after completion of fabrication to maintain cleanliness.

DESIGN REQUIREMENTS FOR SPECIAL SITUATIONS

Reversing Exchangers

Reversing heat exchangers and regenerators are often used in air separation cold boxes. The piping in the reversing streams is subjected to cyclic pressure variations. Fatigue analysis should be performed on this piping. Paragraph K304.8 of Ref. 1 can be used as a guide for the fatigue analysis. Liquid water may be present in the warm-end piping from these heat exchangers. Possible corrosion and freeze-up problems should be considered in the design of this piping.

Liquid Oxygen Evaporation

Most liquid oxygen contains some traces of hydrocarbon contaminants. If this liquid vaporizes in a dead-ended pipe or crevice, the hydrocarbons will concentrate. Explosive mixtures can result. Crevices should be avoided in liquid oxygen piping where a source of heat is present. Drains and other dead-ended pipes connected to a liquid oxygen source should be trapped as close as possible to the liquid source. Liquid oxygen piping should be well-insulated from the casing and from warmer process equipment.

Vacuum-Insulated Cold Boxes

In some cold boxes, a vacuum is pumped on the insulation casing to reduce the heat leak. In such cases, the piping and vessels are tested with helium to find and repair tiny leaks which would greatly reduce the effectiveness of the vacuum insulation. The design pressure for such piping is based on the absolute rather than the gauge pressure.

Instrument Lines

Instrument pipes in the cold box must be designed with care. These pipes have a small diameter and are easily damaged. The design of instrument lines should provide adequate flexibility, support, and protection from insulation and/or inadvertent loads due to using these lines to support the weight of a worker during fabrication. Equation (16), paragraph 319.4.1 (c), Ref.1 can be used for assuring adequate flexibility of instrument lines.

Flammable Fluid

Some cold boxes process flammable fluids such as hydrogen, hydrocarbons, carbon monoxide, and so forth. Any aluminum or other low-melting-temperature piping outside the casing should be protected by fireproof insulation. The insulation should not contain any chemicals which would corrode or otherwise degrade the piping material. Discharges from the drains, vents, and relief valves must be piped to a flare stack or other safe means of disposal. Connections are provided on dead-ended pipes for purging air from the lines before admitting the flammable process fluids.

REFERENCES

1. ASME B31.3, ‘‘Process Piping,’’ An American National Standards, 1996 edition.
2. CGA Pamphlet C-8, ‘‘Safe Practices Guide for Air Separation Plants,’’ Compressed Gas Association, Arlington, VA, latest edition.

LIQUID STORAGE AND CONVERSION SYSTEMS

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Bulk liquid storage systems are often used at or near the point of final consumption. They typically supply product in three basic forms: as a gas at less than the storage tank working pressure, as a gas at high pressure, and as a liquid. The equipment and piping required may vary, depending on which of these delivery forms is used. However, several general considerations apply to the piping design in all cases.

The working pressure of all lines in the system should be at least 10 percent higher than that of the tank. Relief valves required to protect the piping should be set at this pressure. This configuration of relief valve set points will ensure that if the tank is overpressured, it will be relieved in the gas phase from its safety valve and not in the liquid phase through one of the pipeline relief valves.

Vaporizers are commonly used in storage and conversion systems to gasify liquid product and bring it to ambient temperatures. The piping downstream of these units should be designed for liquid product temperature. This should continue up to the point where a safety device is installed to automatically shut off flow in the event that low temperature is sensed. The response time of this device must be compatible with the failure modes of the vaporization system used. The set point chosen must consider the design temperature of all components downstream.

Relief valves should be installed between any set of valves that can trap liquid or low-temperature gas if they are simultaneously closed. These *blocked-line relief valves* are required to relieve pressure buildup due to heat absorption. They should be set at the line working pressure and sized to relieve gas at conditions of maximum heat absorption. Particular attention should be paid to situations where the source of heat is natural convection associated with bare lines and ambient vaporizers. They are of special concern because even moderate winds can greatly increase the rate of heating and subsequent pressure buildup.

Bulk liquid units are typically employed in intermittent flow situations. For this reason, liquid lines will frequently have to be cooled down to liquid temperatures when product is demanded. Therefore, such lines should be sized to pass the required flow in the gaseous state to avoid excessive pressure drop at the start of demand. Design for cryogenic temperature requires that particular attention be paid to line flexibility. Although large displacements are involved due to the extreme temperature changes, they can be accommodated by conventional techniques, including flexible hose sections and expansion loops. In addition, most piping materials experience reduced impact strength at cryogenic temperatures. The materials section in this chapter deals in depth with this topic.

Thermal trapping is a piping technique that can be used in any liquid line, whether it is insulated or not, to minimize heat input to an idle piping section or branch. Where the branch is oriented vertically a gas-to-liquid interface will be established near the connection, and the majority of this section will be filled with gas approaching ambient temperature. This configuration greatly reduces heat input to the branch when it is idle.

LOW-PRESSURE BULK CONVERSION SYSTEMS

The most common bulk liquid units deliver gas at a pressure less than the storage tank working pressure. The working pressure of such systems is typically less than 250 psig (17.2 bar). However, systems with as high as 600 psig (41.4 bar) working pressure tankage have been used commercially. The delivery pressure must be limited to approximately 80 percent of the tank working pressure to allow for tolerance on the safety relief valve setting, and for buildup in product pressure during periods of nonuse. Figure C8.11 illustrates a typical piping configuration, and item numbers in the following discussion refer to this figure.

The lines connecting the vessel the to pressure relief safety devices (No. 301 and No. 302) are sized to meet the requirements of ASME Boiler and Pressure Vessel Code, Sec. VIII, Div. 1. Guidelines for the required calculations are given in the pamphlet CGA S-1.3 ‘‘Pressure Relief Device Standards—Part 3—Stationary Storage Containers for Compressed Gases,’’ published by the Compressed Gas Association. Normally both a relief valve and a burst disk are included. The relief valve is sized to handle the vapor generated due to the heat load resulting from the loss of insulation (loss of vacuum). The bursting disc is designed to handle the simultaneous loss of vacuum and fire conditions. Where it is critical that the system not be taken out of service for periodic inspection of the relief devices, it is common to use a dual set of safety devices with a diverter valve.

The fill system is arranged so that the tank can be filled from either the top or the bottom. Tank pressure is controlled during the fill operation by adjusting the

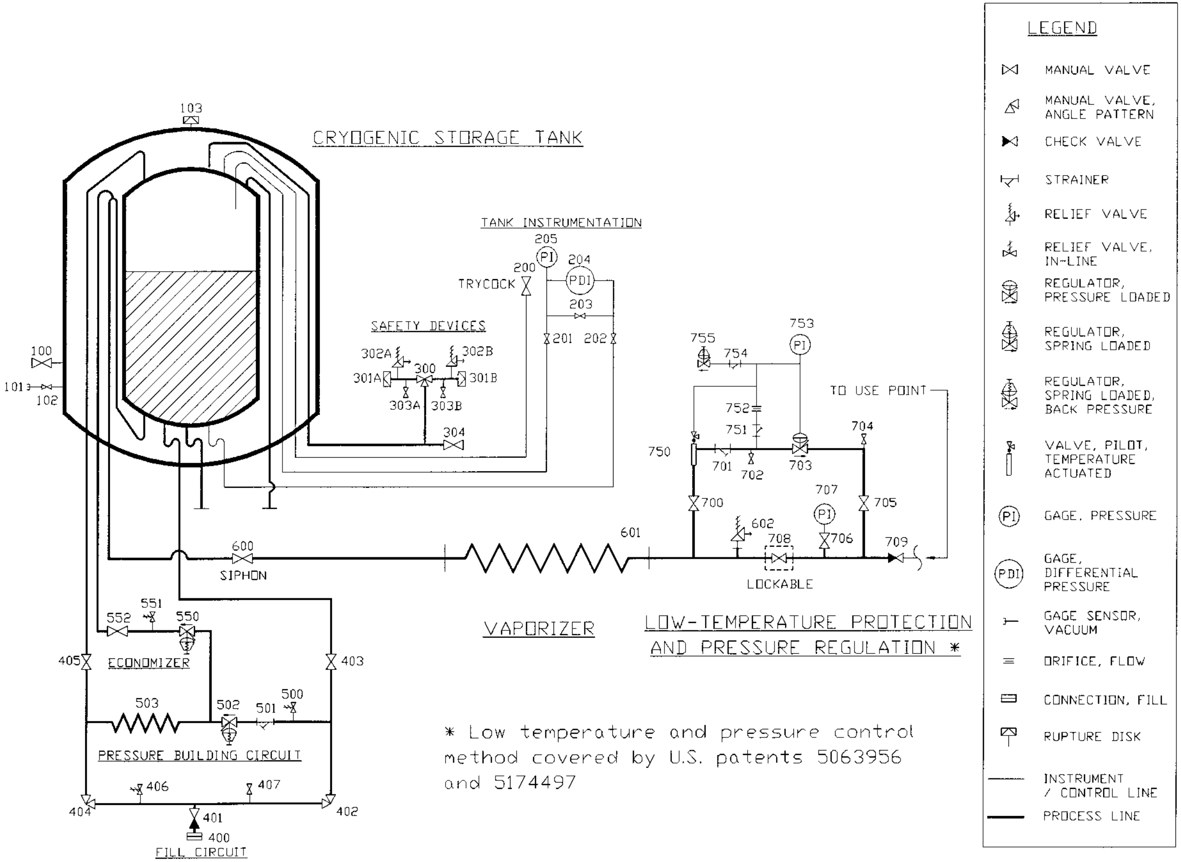

FIGURE C8.11 Low-pressure bulk conversion system *(Courtesy of Praxair, Inc.)*

portion of refill liquid entering from the top relative to that entering from the bottom with valves No. 402 and No. 404. Filling from the top reduces tank pressure by condensing the vapor in the gas space of the storage tank. Filling from the bottom increases tank pressure because the liquid acts as a piston, compressing the gas space. Special piping considerations for the fill lines involve the line size, which should be adequate to permit refill to occur in a reasonable time period and yet not be oversized to limit heat input. Insulation is not typically used on these lines because of their short length, intermittent use, and high transfer rate. Helium and hydrogen fill operations are an exception to this practice because of their extremely low temperature and low heat of vaporization. Other products may justify the use of insulation based on their value.

Bulk tanks are normally filled from transports, which are discussed in the mobile equipment section of this chapter. The transfer is driven either by a centrifugal pump or by pressurizing the supply vessel. The centrifugal pumps are typically one- or two-stage designs matched for the discharge rate and pressure by adjusting the speed and impeller cut as in conventional service. The system—including the transport piping, the off-loading pump or pressurization circuit, the tank fill piping, and the tank pressure relief circuit—must be analyzed to ensure that the tank cannot be overpressurized in the refill process. Compressed Gas Association position paper PS-8, ‘‘Protection of Storage Tanks From Over-pressure during Operator Attended Refill,’’ addresses this topic.

The tank instrument piping includes a gas phase line called the *full trycock* that extends into the gas space to the full liquid level. This line is used by the operator during manual-fill operations to determine when the tank is full. The operator opens valve No. 200 when the fill is nearly complete and observes when liquid is discharged, which indicates the full level has been reached. Also included in the instrument piping is a liquid- and gas-phase line used to measure contents and pressure.

Control of pressure between refills is accomplished by the combined actions of the pressure building, economizer, and pressure-relief systems. The pressure-building circuit consists of components Nos. 500 through 503 and is driven by the difference in densities of the liquid and gas phases. It functions when product flows from the liquid-phase line, is vaporized in the pressure-building vaporizer No. 503, and returns to the gas space. This system is required when the demand for product at the use point is sufficient to cause the tank pressure to fall. Attention must be paid to the very low driving pressure in this circuit.

Product is normally delivered to the use point from the tank in liquid phase. In the case of low or intermittent demand, the product vapor pressure can increase above the delivery pressure. At this point pressure-building is no longer necessary, and it is desirable to withdraw product from the tank in gas phase. Product delivered in the gas phase will remove approximately five times the heat from the tank as the same mass withdrawn in liquid phase. A scheme to automatically switch to gas phase supply at high tank pressure is called an *economizer*. A siphon cycle economizer is illustrated in items Nos. 550 through 552, together with the siphon line inside the tank. When tank pressure is high, regulator No. 502 is closed and back-pressure valve No. 550 is open. The siphon is broken in this way, and gaseous product is withdrawn through valve No. 600. The siphon is reestablished at reduced tank pressure when the positions of valves Nos. 502 and 550 are reversed. There are alternative economizer designs, and care must be taken in selecting one because most are limited to low instantaneous flows.

The product is next vaporized and superheated in the vaporizer No. 601. Vaporizers are classified as *ambient* where the required heat is derived from the atmosphere, or as *powered* where the heat is supplied from another source such as steam, electricity, or hot water. Relief valve No. 602 provides protection in the event of a blocked line condition, and special attention should be paid to the vaporizer power source in selecting this valve.

A low-temperature pipeline protection device is normally included downstream of the vaporizer to automatically shut down flow in the event of vaporizer failure. This shutdown device is the transition point between cryogenic and noncryogenic pipe design. In addition, a pipeline regulator is usually used to provide constant pressure in the pipeline. The low-temperature protection and pressure regulation module given as components Nos. 700 through 755 has emerged as the preferred way of providing these functions. Regulator No. 703 is pressure-loaded so that discharge pressure is approximately equal to the loading pressure as read on gauge No. 753. Temperature pilot No. 750 opens and reduces the loading pressure when gas temperature falls into the set control range, until No. 703 is completely closed.

HIGH-PRESSURE BULK CONVERSION SYSTEMS

In some applications, gas is required at greater than the working pressure of the tank. In such cases, it is pumped in the liquid state, vaporized, superheated to ambient temperature, and regulated to the required pipeline pressure. A bank of high-pressure receivers is usually included to match the demand to the pump discharge rate. The pump is typically a reciprocating single-acting design ranging in configuration from simplex to triplex. Figure C8.12 gives a typical flow diagram of a high-pressure conversion system employing a standard cryogenic storage tank. Item numbers referred to in the following discussion are given in this figure. Piping practice for much of the system remains unchanged from the low-pressure gas system just discussed. However, special considerations must be made for the piping both upstream and downstream of the pump to ensure proper system operation.

The fluid at the suction flange must be subcooled in order to meet the net positive suction head (NPSH) requirements. The degree of subcooling required ranges from 2 to 10 psi, depending on pump design, condition, and discharge requirements. The liquid at the bottom of the storage tank is normally subcooled by approximately the static head of the liquid phase above it. The suction piping must minimize both heat input and pressure drop to preserve the available subcooling. Pressure losses are primarily due to the fluid acceleration required by the reciprocating pump and may be calculated using classical analysis. Heat input generally increases with pipe diameter, but may be essentially eliminated by the use of high-performance insulation. Insulation should always be used where the tank working pressure is low, the demand is intermittent, or the pump discharge rate is low. It is used almost universally in hydrogen pumping systems.

The pump is usually contained in a small vacuum-insulated sump, which is connected to the storage tank gas and liquid phases. As the first step in priming the pump, components Nos. 800 through 807 are used to fill the sump with liquid. The product vaporized in cooling these components is returned to the tank gas phase, increasing system pressure and thereby available subcooling. The second step is to run the pump with valve No. 827 open, which circulates liquid through the pump and discharges it to the tank gas phase, cooling the pump compression chamber. Finally, valve No. 827 is closed, forcing the pump to discharge against receiver pressure which is being held by check valves No. 830. Temperature sensor No. 831 is used to check for pump prime.

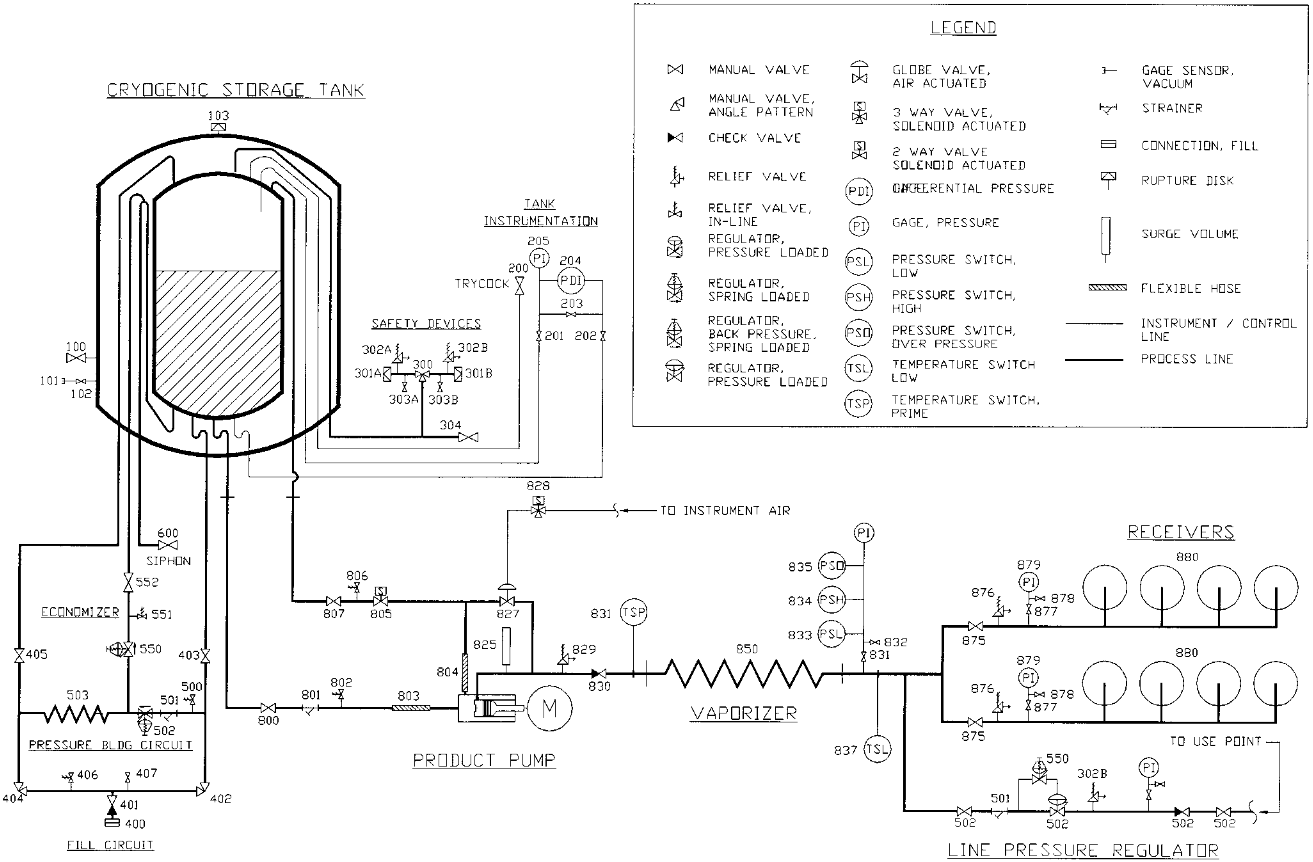

FIGURE C8.12 High-pressure bulk conversion system. *(Courtesy of Praxair, Inc.)*

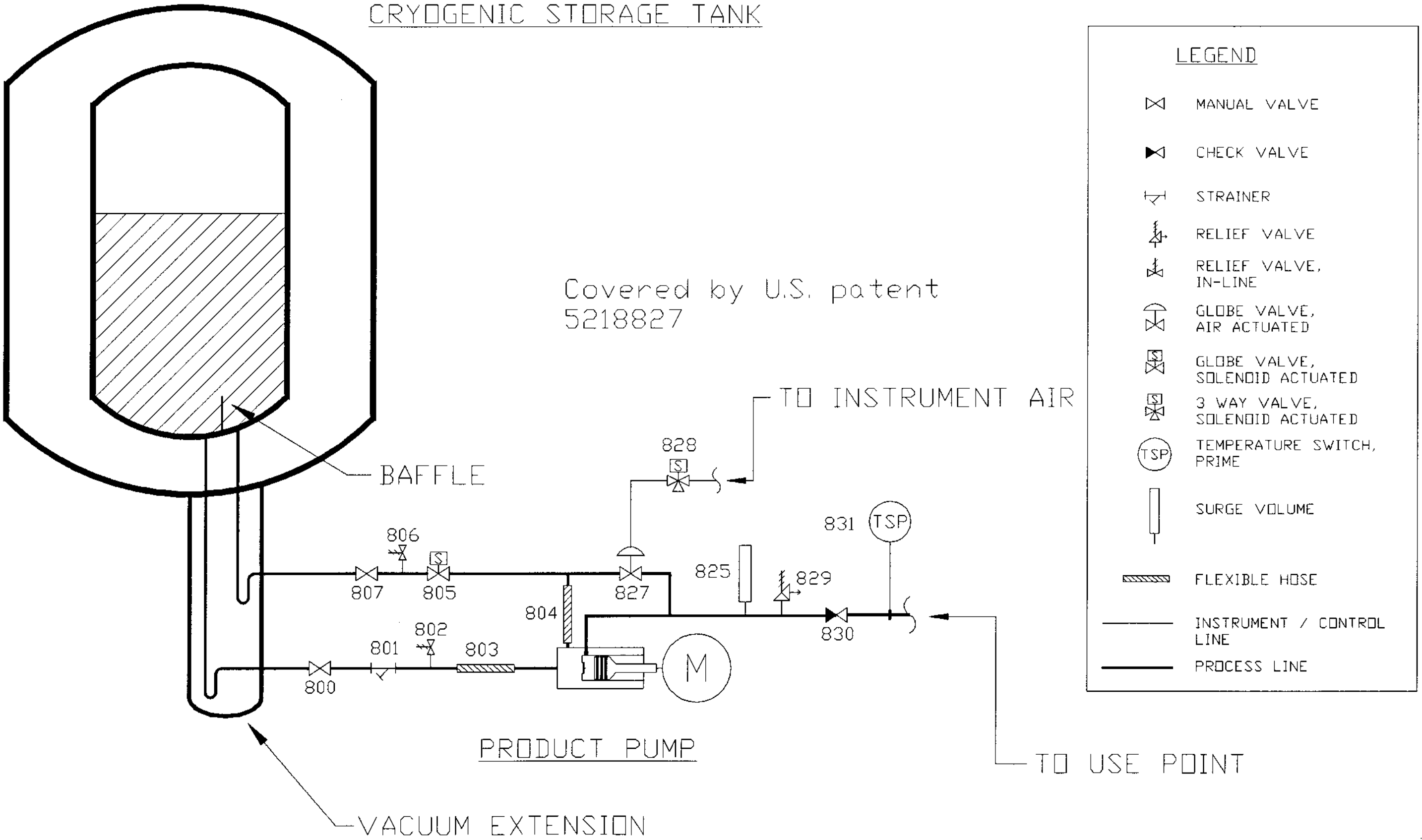

FIGURE C8.13 Thermosiphon prime system for cryogenic pumps. *(Courtesy of Praxair, Inc.)*

The piping downstream of the pump must be designed for cryogenic temperatures and high pressures as well as the pulsations and vibration generated by the reciprocating pump. Temperature switch No. 837 is used to shut the pump off in the event of low pipeline temperature. Although noncryogenic materials may be used downstream of this sensor, it is common practice to use cryogenic materials for all high-pressure piping in the bulk delivery system.

Priming with this conventional piping arrangement works well while the liquid level in the tank is high. In this case a circulation flow develops, with fluid gaining heat in the path through the suction line, the pump sump, and the return line, which reduces its density. The lightened liquid flows up the return line and dumps into the tank. This circulation is driven by the difference between the static head due to the tank gas and liquid phase contents and that due to the less dense liquid in the return line. When the depth of the liquid contents is nearly equal to the height of the tank, this circulation flow quickly cools the pump and presents subcooled liquid at the suction valve to enable a good pump prime. However, circulation flow is reduced at lower tank levels because the static head of the tank contents is less, and priming becomes impossible. Figure C8.13 gives an improved arrangement in which both the suction and return lines are connected to the tank liquid phase through an extension of the vacuum jacket. The two penetrations are separated by a short baffle to promote convection within the tank liquid phase. The difference in height between the traps within the extension provides the head needed to start the circulation flow. The heat picked up in traveling to and from the pump sump reduces the density of the returned liquid, which maintains the circulation once started. This configuration allows priming to occur regardless of the tank level.

BULK LIQUID DELIVERY SYSTEMS

Where liquid-phase product is required at the use point, refrigeration rather than the fluid itself is usually of interest. Therefore, minimizing heat addition through the tank and piping is the primary design consideration in these systems. Some degree of pipe insulation can almost always be justified. The tank insulation performance is also critical to satisfactory system operation.

Ideally, the tank should be elevated so that the entire pressure requirement can be provided by the static head of the tank contents. This eliminates the need for pressure building in the storage tank, which can easily represent several times the heat load from all other sources. A horizontal tank is preferred to a vertical tank, in this case, both because the elevated foundation is less expensive and because the variation in static head, as the contents are withdrawn, is reduced. Where pressure building is required and the tank will not be elevated, a vertical tank is preferred to minimize heat transfer through the gas to liquid interface area of the vessel.

In spite of the efforts, the fluid normally arrives at the use point with at least a small gas component. Normally, conditioning by a subcooler, phase separator, or similar equipment is required to achieve satisfactory process control. This hardware is located at the application and at a minimum delivers saturated liquid by venting the gas that is generated in the piping system.

Figure C8.14 illustrates a self-contained system that delivers subcooled fluid. It consists a of heat exchanger formed by a tube within a tube, called an *in-line subcooler* because it replaces a section of the supply line. It is arranged so that the

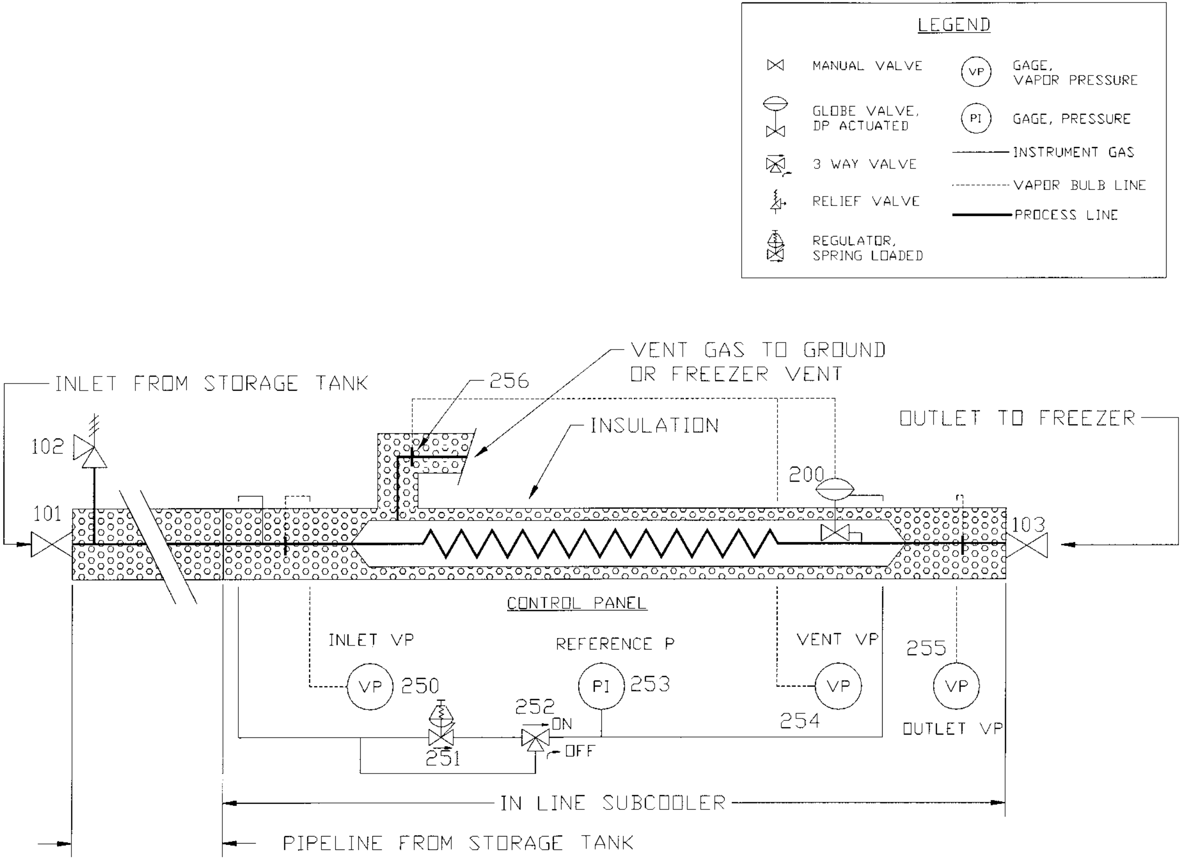

FIGURE C8.14 Inline cryogenic subcooler. Courtesy of Praxair Inc. (*Covered by U.S. Patent No. 5477691.*)

main product stream flows through the inner tube, and control valve No. 200, located at the discharge end, emits a small portion into the annular space. This control valve is actuated by the difference between the vapor pressure of bulb No. 256, located in the vent, and the reference pressure set by regulator No. 251. When the vapor pressure of the vent fluid rises above the reference pressure, the control valve opens. The reference pressure is selected to ensure that cold gas—but not liquid—is emerging from the vent. The temperature of the liquid in the annular space is reduced, since the pressure is maintained at a near-atmospheric level. Liquid flowing through the inner tube is cooled by the annular stream.

MOBILE EQUIPMENT SYSTEM

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Bulk atmospheric gases, oxygen, nitrogen, and argon are transported as liquids in various styles of double-walled tank trucks. This section presents the most common semitrailer-style transport vehicles used for the distribution of atmospheric gases.

A doubled-walled cylindrical tank truck has a product tank, or inner pressure vessel, enclosed in an outer casing or jacket. The inner pressure vessel is designed, manufactured and tested to meet requirements of Sec. VIII of the ASME Boiler and Pressure Vessel Code. The casing or jacket is designed to the requirements of CGA-341, Standard for Insulated Cargo Tank Specification for Cryogenic Liquid, Compressed Gas Association. The annular space between the inner pressure vessel and casing is insulated and evacuated in order to minimize the amount of heat leak from the casing shell to the cryogenic liquid. Heat transferred to the product will result in a pressure rise, with the eventual release of product. The inner pressure vessel is normally supported or attached to the casing at or near the inner pressure vessel heads. The upper coupler king pin, landing gear support legs, and tandem axle assembly for the cryogenic semitrailer are standard automotive components available to the trucking industry.

Atmospheric gases are typically transported in low-pressure semitrailers equipped with an on-board pumping system used for off-loading the product to a customer storage tank. This pumper-style semitrailer allows for a maximum payload at a gross combination weight limit of 80,000 lbs (36,300 kg)—the road limit for a five-axle tractor-trailer combination. Inner pressure vessels are rated for a maximum allowable working pressure of from 40 to 60 psia (276 to 414 kPa) and constructed from AISI type 304 stainless steel or AA-5083 aluminum alloy (ASTM B-209). Casings are constructed from either carbon steel or aluminum alloys. Carbon steel casings are used along with stainless inner pressure vessels, and aluminum casings are matched to aluminum inner pressure vessels. A lightweight casing design can be achieved by providing closely spaced circumferential ring stiffeners. These ribs reinforce the cylindrical casing section against the external pressure load which results from the evacuation of the insulation or annular space.

Atmospheric gases have different product densities, which will in turn result in different-sized product-optimized semitrailers. When optimized for an 80,000 lb [36,300 kg] road limit, a nitrogen-sized semitrailer has a volume of about 8000 gal (30.3 m^3); an oxygen semitrailer about 6000 gal (22.7 m^3); and an argon semitrailer about 4500 gal (17.1 m^3).

Liquid oxygen, nitrogen, and argon are exempt from the U.S. Department of Transportation (DOT) hazardous material regulations for design requirements of the semitrailer or package when the product pressure is maintained at less than 40 psia [276 kPa] during transportation. However, DOT markings and shipping preparation, along with regulations pertaining to the trucking components such as braking and lighting systems, are all considerations when designing a cryogenic transport. Basic requirements for vessels, structural supports, structural protection, and external piping are covered by CGA-341 specifications.

Piping systems for cryogenic transports can be separated into two categories: *internal piping,* which includes piping internal to the inner pressure vessel as well as annular space piping; and *external piping,* which includes all piping exterior to the casing envelope. Both internal and external piping systems are normally fabricated with butt-welded joints.

INTERNAL PIPING

An internal piping system is constructed from stainless steel for stainless inner pressure vessels and of aluminum for aluminum inner pressure vessels. A typical internal piping arrangement has the piping penetrating the vessel walls through the rear casing head, as compared to a location on the cylindrical portion of the vessel. (See Fig. C8.15 for a typical piping schematic.) Piping sections inside the inner pressure vessel are not subject to an internal pressure loading. Design

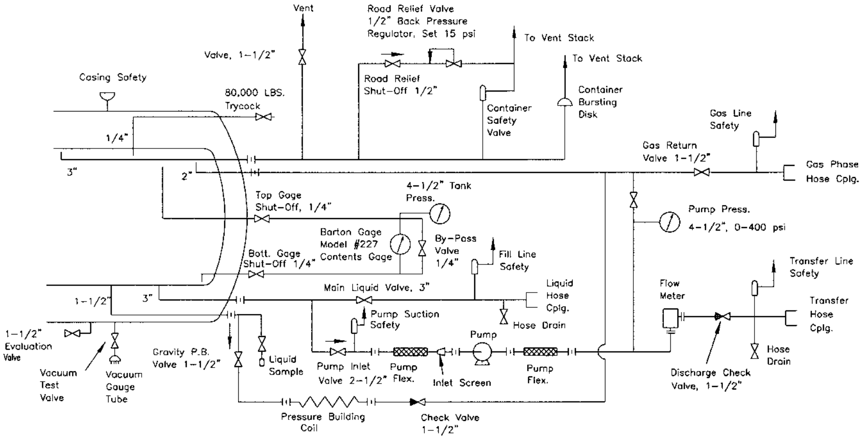

FIGURE C8.15 Schematic of typical internal piping arrangement.

considerations for piping supports and attachments include loadings imposed by road vibration and thermal contraction due to temperature as low as −320.4°F (−195.8°C).

Annular space piping configurations must have consideration given to heat leakage, flexibility, and trapping of liquid or gas. Since heat leakage to the cryogenic liquid is primarily by conduction down the pipe wall, the length of the piping run is of importance. Stresses induced by thermal contraction of the line or movement of the inner pressure vessel relative to the casing are typically accommodated by providing adequate piping flexibility in the form of loops and bends. Stress levels due to flexibility are generally kept to within the allowable limits imposed by ASME B31.3, Process Piping. Piping bellows to accommodate thermal displacements are not generally considered an acceptable way of providing flexibility to annular space piping. Road vibrations have proven to be detrimental to bellows-style fittings.

Testing of internal piping systems includes pressure, leak, and other examinations when required by codes. Pressure-testing procedures follow ASME B31.3 requirements. Nozzle penetrations at the inner pressure vessel wall may require examination of the welded joint using dye-penetrant techniques. Annular space piping is thoroughly leak tested, since any leaks would deteriorate the vacuum condition within the insulation space. A helium mass spectrometer technique is the most common method used for vessel and piping leak testing.

EXTERNAL PIPING

The external piping system is commonly located inside an operating compartment at the rear of the semitrailer; however, the pressure-building coil is located under the semitrailer forward of the tandem axle assembly. The rear grouping has accident protection features provided by the tandem axle subframe, rear bumper, and the piping compartment.

Stainless steel external piping systems are used on all transports regardless of inner vessel construction, with the exception of the pressure-building coil. The coil is normally constructed of aluminum extruded-fin tubing, which is flanged to stainless steel inlet and outlet piping. The function of the coil will be discussed later. Valves, gauges, and other instruments and controls are not necessarily constructed from stainless steel materials. Bronzes, copper alloys, and aluminum alloys are used for various component parts. Internal and external piping runs are joined at the casing penetrations with piping flanges. For aluminum vessel designs these flanges provide a simple way of connecting aluminum internal lines to stainless steel external lines.

As Fig. C8.15 schematically illustrates, cryogenic semitrailers typically have two liquid phase lines. The first piping run is used to fill the trailer at the production plant and to unload the trailer using the off-loading pump. The second liquid line is used for the pressure-building system. Pressure in the inner pressure vessel can be increased by vaporizing a small amount of liquid. The coil with a large heat transfer surface can readily vaporize liquid and return the warmed gas to the ullage space of the trailer. This pressure buildup is performed prior to and during the off-loading process in order to maintain adequate suction pressure for the centrifugal transfer pump.

Gas-phase piping includes a pressure-relief device line which directly communicates with the vapor or gas space near the midpoint of the top centerline. A springloaded pressure relief valve and a rupture disk device are normally provided on the cryogenic semitrailer. These relief devices are designed to maintain pressure at a safe level under emergency conditions, including exposure of the vessel to a fire. Pressure-relief devices are designed to the requirements of Section VIII of the ASME Boiler & Pressure Code as well as CGA pamphlet S1.2, ''Pressure Relief Device Standards—Cargo and Portable Tanks for Compressed Gases.'' The same piping run is also used to provide for a manual vent system and a *road relief* circuit. The road relief valve, typically set at about 30 psia (207 kPa), controls the inner vessel pressure during transportation. Venting of product when the pressure reaches the road relief valve setting can occur. A shutoff valve upstream of the road relief allows isolation during off-loading and, therefore, permits the buildup of pressure to an operating level near the working pressure of the inner pressure vessel.

A second gas-phase circuit has multiple uses as a gas-phase outlet, pressure-building coil return, and a transfer pump recirculation line.

An optional sparger, or gas-phase fill header, has various uses. A typical design has a pipe running along the top of the inner vessel with a series of holes which act as spray nozzles for the liquid. With this line teed from the main fill line, transfer of liquid to the trailer can occur in an all-liquid phase, all-gas phase, or any combination. Gas-phase filling of an empty trailer through the sparger will cause a partial collapse of the gas pressure. This pressure reduction by collapse eliminates product losses that would occur by manually venting the pressure from the vessel. A sparger also provides a means of uniformly cooling down or shrinking a warm inner pressure vessel. Uneven shrinking of a inner vessel from near ambient to liquid temperatures can cause serious structural damage to the vessel, supports, or piping.

Inner vessel pressure, transfer pump discharge pressure, and liquid contents are monitored with trailer-mounted gauges. A differential pressure indicator is the most common device used for contents measurement. Liquid and gas taps are provided for this gauge.

Sampling of product in the inner pressure vessel is necessary in order to determine the level of product purity. A liquid tap typically teed externally to a liquid line can be used for sampling.

The liquid fill level can be detected by a small-diameter fixed-length dip tube. Flow of liquid through the line indicates the liquid level in the tank. If the semitrailer is used to transport various cryogenic liquids, dip tubes positioned at different levels would be provided.

As previously stated, the annular space is evacuated. Pressure levels below 0.04 in (1×10^{-4} meter) of mercury are achieved by pumping down the space through a line equipped with a shutoff valve. A filter on this pipe eliminates problems with the insulation material being carried to the valve or pump. The vacuum level can be monitored using the trailer-equipped thermocouple gauge tube. Vacuum level is determined by a portable vacuum meter.

Cryogenic piping must be protected from overpressurization in any piping section which can be isolated. After liquid or gas has been transferred, the cold product warms up and builds pressure which can exceed the bursting pressure of the pipe or fittings. For this reason, *block line safety devices* are a mandatory feature in cryogenic piping circuits.

Thermal contraction of external pipes can cause high forces when constrained. Typically, centrifugal transfer pumps and meter systems are protected from these forces by expansion bellows in the inlet and discharge piping. Piping bellows are made from corrugated stainless steel hose.

ULTRA-HIGH-PURITY PIPING

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Ultra-high-purity (UHP) piping systems are found in gas and liquid supply systems used in the fabrication of electronic semiconductor chips and other related devices. The requirements for purity in UHP systems typically involve contamination levels of less than 1 part per billion (ppb) of foreign materials.

Semiconductor fabrication involves forming alternate layers of very fine lines of semiconducting material on the surface of nonconducting material such as silicon. The lines form miniature ultra-large-scale-integrated (ULSI) electronic circuits on a small silicon chip. Many small chips are simultaneously fabricated on a single silicon wafer. The line or feature size of ULSI chips approaches 5 μin (0.13 μm) or less. Any particle one-tenth of the line size can cause a defect in the circuit, resulting in chip destruction. To keep the percentage output yield of defect-free chips high, the gases and liquids used in the fabrication of ULSI circuits must be very pure and free of particulate contamination.

The present requirements for gas purity are that trace gas contamination must be held to the parts-per-trillion (ppt) level. For particulates, contamination with particles greater than about one-tenth the semiconductor feature size must be held to less than 5 particles/ft^3 (190 particles/m^3) of gas.

GENERAL GAS SUPPLY SYSTEM DESCRIPTION

Semiconductor manufacturers require UHP gases at the point of use inside their facilities (fabs). In most industries, a gas supplier is only required to supply gas to some interface point near the customer's plant boundary. However, for ultraclean systems, the gas supplier is typically required to provide UHP gas to the final point of use. This means that the gas supplier is responsible for the UHP piping system within the customer's fab, in addition to the gas storage and conditioning system at the fab boundary. This ensures that the gas meets the customer's requirements at the point of use.

Gas supply systems can range from single small cylinders to large air separation plants. The specialty gas requirements can usually be met with high-pressure gas cylinders, although bulk specialty gas supply systems are becoming increasingly more common. Bulk gas supply requirements are usually met by a combination of on-site production facilities and liquid storage pad packages. However, regardless of the gas supply system size or complexity, it must be able to reliably supply gases in UHP condition to the customer.

Typical facilities require most, if not all, of the supply systems described above. High-volume consumption gases such as nitrogen and clean dry air (CDA) are usually supplied from an on-site plant. A typical on-site system may consist of air

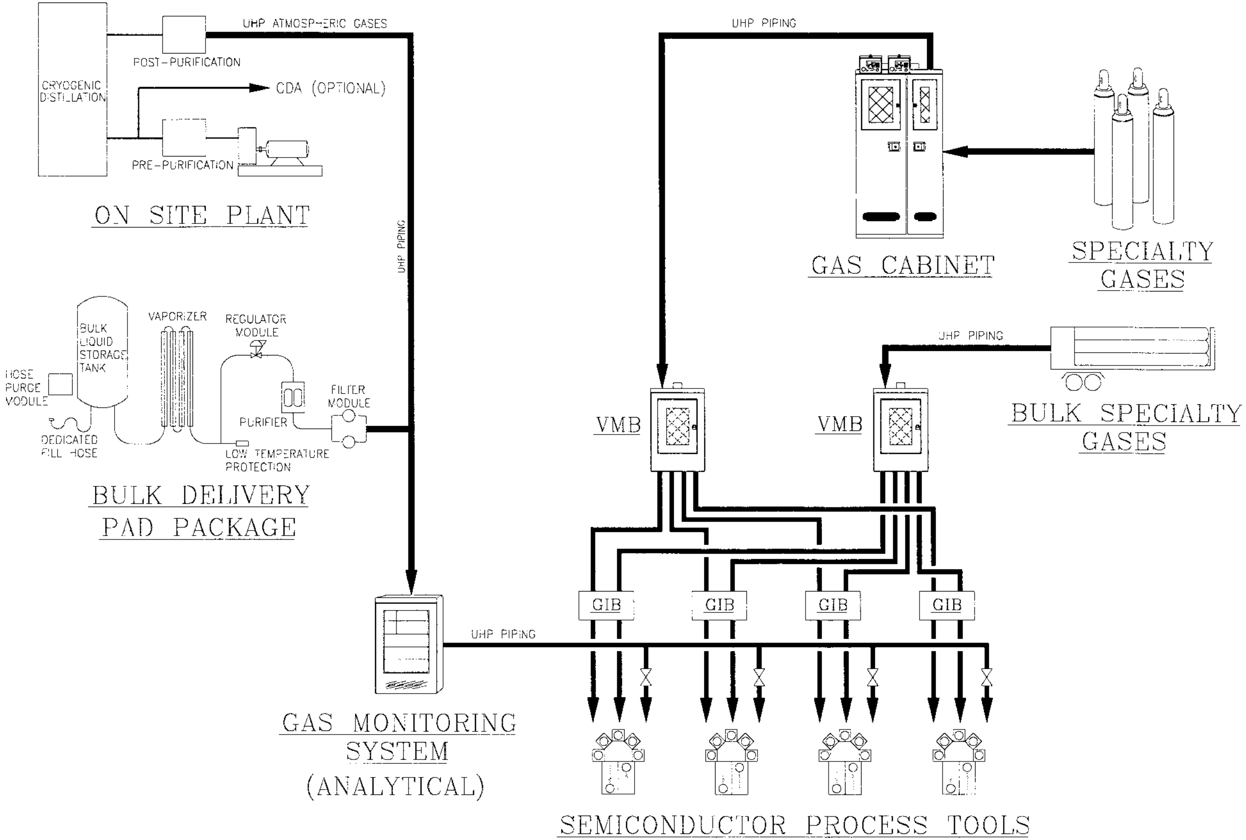

FIGURE C8.16 Ultra-high-purity gas supply system. *(Courtesy of Praxair, Inc.)*

compressors, a cryogenic distillation column, purification equipment to remove selected impurities from the air or product streams, control systems to regulate flow and pressure of products delivered to the customer, filtration systems to remove particulate contamination, and a piping system to deliver the gas to the customer's point of use.

Other bulk gases and backup supplies for nitrogen are supplied from bulk liquid storage pad packages. These systems typically consist of a bulk liquid storage tank, a vaporizer to convert the liquid to gas, control systems to regulate the flow and pressure delivered to the customer, gas purifiers to remove any residual trace gas impurities, filtration systems to remove particulate contamination, and a piping system to conduct the gas to the customer's point of use.

In some cases, liquid supply is needed also. Usually the liquid is transported to the point of use in a small storage vessel or dewar. For some supplies, the liquid may be piped directly to the point of use using UHP liquid vacuum-insulated piping (VIP). In these instances, the inner carrier pipe of the VIP must meet the same requirements as the UHP gas piping.

Most specialty gas supply systems are from special high-pressure, high-purity cylinders, or from bulk specialty gas supply systems if their usages are high. Bulk specialty gas supply systems replace the high-pressure cylinders typically used with larger volume containers such as high-pressure modules or tube trailers or special bulk containers for liquefied specialty gases. Specialty gas supply systems typically consist of the gas source (cylinder or bulk) with the appropriate gas cabinet or gas panels with controls to regulate the flow and pressure to the customer; valve manifold boxes (VMBs) or panels to distribute to multiple use points where appropriate; gas isolation boxes (GIBs) to isolate sources of gas at a point of use; and interconnecting piping. Piping may be single-wall or double-contained, depending on gas characteristics and local codes and regulations. These installations may include purifiers where appropriate.

A schematic representation of a typical supply system to a fab is shown in Fig. C8.16.

SYSTEM DESIGN AND FABRICATION REQUIREMENTS

UHP gas supply systems must meet extremely stringent design, fabrication, installation, checkout, and commissioning requirements. A brief description of typical requirements is presented in this subsection.

Design Requirements

Designers of UHP gas systems need to consider many items often thought to be unrelated to the piping design. Obviously, the designer must consider the basic system requirements such as flow and pressure. Basic material compatibility within the system and especially with the gas itself must also be considered, since many of the gases are toxic and/or corrosive and/or flammable. But the designer must also look at the entire facility design philosophy and be familiar with the basic safety considerations, codes, and regulations. What quantities of gases will be permitted on-site for use and for storage? How will hazardous gas monitoring and toxic gas detection be handled? Answers to these questions may alter gas systems design, determine which systems are to be double-contained, and how the containment system is to be installed and monitored.

Once these considerations are resolved, then the system designer can proceed and address such things as purity and contamination issues. Traditional industrial gas systems are considered clean when contamination levels meet parts-per-million (ppm) requirements. In the semiconductor industry, however, contamination levels must meet purity requirements on the order of less than 1 ppb. For instance, particulate cleanliness may require the following specifications:

$$< 10 \text{ particles/ft}^3 \geq .01\mu\text{m in size} \quad (< 380 \text{ particles/m}^3 \geq 0.4\ \mu\text{in in size})$$

$$< 1 \text{ particle/ft}^3 > 0.1\ \mu\text{m in size} \quad (< 38 \text{ particles/m}^3 > 4\ \mu\text{in in size})$$

Trace gas impurities are typically trapped within parts of the system during component manufacture and system assembly, and later released into the process stream. Joining materials, laps within joints, screw threads, and other cavities can trap moisture, welding gases, and other contaminants and can become long-term outgassing sources, often referred to as virtual leaks. For this reason, such geometric discontinuities must be eliminated from the system by design and through proper fabrication and installation practices.

Particulate contamination is found in many of the same places as the trace gas contaminants (i.e., crevices, joints between mating pieces, dead-end legs off the process stream). In addition, particulates become trapped within indentations and small voids that exist within the natural roughness of most materials. For this reason, all pipe and component surfaces exposed to process gas streams are polished as smoothly as possible to eliminate contamination from this source. Industry standards typically require surface finishes to be electropolished to a value of less than 7 μin Ra (0.28 μm Ra) roughness. Electropolishing also leaves an enriched chromium oxide layer on the surface. Alternative surface treatments such as special passivation techniques are sometimes used in selected system designs, especially for corrosives.

Particles can also be generated by erosion of the process stream over surfaces of system components in the flow path. Also, contact between moving parts scrapes off metal particulates from contacting surfaces. Therefore, sharp edges, rough surfaces, and sliding or contacting surfaces must be eliminated from the design.

Many process conditions promote the generation of particulates or promote the release of particulates from component surfaces. High process gas velocities erode particulates from component surfaces. Vibration or mechanical shocks shake loose particulates resting on process flow surfaces. These actions can either be caused by external conditions such as adjacent rotating or reciprocating machinery that is not properly dampened or isolated from the system, or from valve or regulator operation within the system. Valve or regulator operation may also generate particles resulting from contact and erosion of moving surfaces.

Finally, transient process conditions can also generate contamination. Variations in system temperatures may enhance release of trace-gas impurities or cause thermal expansion and contraction that could cause particulate release. Outgassing and particulate shedding upon system start-up can also release contamination into the process stream.

The basic design concept in UHP gas systems design is to *design clean, build clean, and operate clean*. The UHP gas systems should be designed to be leak-free, outgas-free, deadspace-free, and particle-free. Component selection should include a thorough evaluation of all the components in static, dynamic, and impacted conditions.

Materials of Construction

The obvious starting point in meeting requirements of this type is to use the right materials. All materials must be compatible with the process gas itself in order to avoid such problems as corrosion. In addition, all materials in the system must be compatible with each other to avoid chemical or electrical reactions between different materials and again to prevent corrosion problems.

The only material generally acceptable for fabrication of bulk gas piping and system components is 316L (low carbon) stainless steel. In addition, the steel must have low sulfur content in order to obtain better weld qualities. Where 0.035 percent sulfur content is acceptable for normal applications, the sulfur content for UHP systems should be in the 0.003 to 0.017 percent range.

As the semiconductor industry continues to reduce feature size and line widths, component and tubing manufacturers have to evaluate and address ever-increasing concerns over not just the sulfur content, which impacts both weldability of the system and machinability of the component, but also such concerns as nonmetallic inclusions in the base steel. This has led to the increasing use of vacuum arc remelt (VAR) or VIM/VAR and other specially produced 316L stainless steels.

Additionally, gas-absorbing materials (especially nonmetallics) must be avoided. Contaminant gases dissolve into many materials of construction. This occurs primarily in nonmetallic components such as valve seats and filter cartridges, but it can also occur in the metallic parts. Since so much steel exists in the system components, dissolved gases within these components could present a serious contamination problem. Special manufacturing procedures, as well as final cleaning and conditioning procedures, must be considered to reduce dissolved gases within steels and other metals used in ultraclean systems.

Many nonmetallic materials, particularly those used in the filter cartridges, tend to dissolve gases, primarily water vapor. This is one of the most serious contaminants in semiconductor fabrication. Special materials or special procedures to remove the contaminants are necessary to prevent these contaminants from entering the system.

Any material not used in the particular semiconductor fabrication step could cause unacceptable contamination. Heavy-metal ions cause severe problems when they enter the semiconductor fabrication process. Metals such as sodium used in the fabrication or cleaning process of other metallic parts could leave undesirable metal ions on or within the parts. These ions must also be eliminated as much as possible.

Ideally, all pipe and components for one piping run should be of a single heat. This ensures that chemical properties of the two pieces at a weld joint are similar. In practice, this is often impossible. Most important is to ensure that all tubing and components have similar chemistries to guarantee weldability. If it is necessary to use a component with a dissimilar chemistry, care must be exercised to ensure weldability and integrity.

Standard elastomeric seal materials such as Teflon®, Kel-F™, etc., are acceptable for seals. However, if metallic seals can be designed into the system, greater leak-tight systems are possible. For this reason, valves with diaphragm or bellows seals on the stems should be used to totally isolate the product stream from external contamination.

Purity and cleanliness are important. Outgassing of dissolved or adsorbed gases must be reduced to very low levels. This is sometimes accomplished by special material processing such as passivating steels in acid mixtures or by subjecting steels to a double-melt process. It may be necessary to ‘‘bake out’’ some of the components or the entire system, if the contamination is serious, to drive off the adsorbed gases.

Welding

Conventional welding practices such as lap and socket welding are unacceptable for UHP application since they do not permit full penetration and leave overlaps and crevices that are very difficult to clean, creating virtual leaks. Hand welding, which leaves rough surfaces on the interior, is also unacceptable. Rough surfaces, as well as discoloration from the heat-affected zone, are sources of particulate generation. Rough surfaces also retain more moisture.

All process line tube joining is done by autogenous orbital butt welding performed by special automatic process welding machines. The joint to be welded is purged both internally and externally with high-purity inert argon to avoid oxidation of the hot weld surface. A good orbital weld must be free of stain on the product surface, can have no undercut or concavity of the weld area, and must have a generally smooth process surface finish. Weld buildup and metal puddling are not acceptable.

Weld samples (or coupons) must be made at the beginning and end of each shift or whenever the weld machine variables or parameters such as line size or heat number are changed. These weld coupons must be marked with the welder's name, and a log of all sample welds must be maintained.

Weld requirements for field welds are the same as for shop welds. That is, all welds must be autogenous orbital welds wherever possible. All welding and weld preparation takes place in either a clean room or a controlled work area to maintain system cleanliness. Field welders must pass the same certification requirements as shop welders.

Fabrication and Installation

Fabrication and installation procedures for UHP systems are not dramatically different from normal industrial practice, except for cleanliness. Because of such similarity, cleanliness becomes analogous to quality or safety in that it involves a culture rather than a procedure. Procedures are easy to establish; cultures are not. A culture requires a completely new approach to the fabrication and installation process.

Fabrication of UHP gas supply systems must be conducted in as clean a manner as possible. The work should be done in an enclosed shop where clean conditions can be controlled more easily. Generally, it is less expensive to build as much as possible in the shop. To provide the cleanest conditions possible, all component fabrication must be conducted in a clean room, preferably at least Class 100 quality, as determined by federal standards.

The UHP gas systems must be fabricated as a system. Each step in the fabrication and installation process involves UHP argon purges. Once the fabrication process begins, the entire system should be kept under a UHP argon purge until the system is ready for introduction of the process gas at start-up.

Since cleanliness is a prime requirement, the cleaning process is one of the most critical steps in all stages of fabrication and assembly. As stated earlier, in addition to general cleaning, cleanliness involves polishing (i.e., mechanical, chemical, or electropolishing). All parts must be cleaned at critical steps in the manufacturing process and must be kept clean between fabrication steps and during shipping.

The best method of cleaning is to electropolish the component. This procedure actually removes some of the surface metal, along with any contaminants on the surface.

All gas supply system components such as valves and regulators should be fabricated in a shop where cleanliness can be controlled. Also, some subassemblies and piping ‘‘spools’’ should be fabricated in a cleanliness-controlled shop area. However, fabrication of the gas distribution piping to interconnect the various components is usually done in the field at the job site. This exposes the components and piping pieces to contamination from the atmosphere. Therefore, the division of work between the shop and the field must be carefully considered.

Components must be packaged to ensure the cleanliness level is maintained. Double-wrapping in plastic bags is a minimum requirement. Piping must have the ends capped. The tubes must be pressurized with pure nitrogen or argon, or sealed in a pressurized container. Consideration must be given to both the final destination and the route of transit. Changes in altitude, and hence pressure, could damage packaging.

Once the clean components arrive at the plant site, they must be stored so that the cleanliness of the components is maintained. While waiting for installation, all components must be stored in their protective wraps, and only opened when ready to be finally installed.

Special care and procedures must be used to install the component without introducing unnecessary contamination. It may be necessary to provide a portable clean room at the installation site to perform some of the required on-site fabrication in as clean an environment as possible. The clean room should be at least Class 100 quality. As much of the subcomponent assembly as possible should be done in this portable clean room. After subcomponent fabrication, all open ends of ‘‘spool pieces’’ must be covered with an anti-static polyethylene or anti-static mylar wrap and kept sealed until just prior to welding into the final assembly.

It may also be necessary to construct a clean environment around some system parts to ensure that minimum contamination is introduced as final component installation is performed.

To reduce the possibility of atmospheric contamination entering the partially fabricated piping system, the portion of the system that has been completed must be continually purged with a pure, inert gas filtered to submicrometer levels. Since inert gases are used for purging and for maintaining clean conditions, safety provisions must be made to protect personnel against asphyxiation.

In addition, all tools that might come in contact with process gas surfaces must be maintained in as clean a condition as possible. These tools must be specially cleaned; used only on the clean system; and stored in the clean room or a clean environment.

QUALITY ASSURANCE

An independent quality assurance representative should be used to review and approve all installation procedures for the construction of a gas supply system, and to conduct certain tests and inspections as agreed to by the owner. These procedures and tests should include the following activities:

1. Inspect the precleaned, UHP components prior to shipment.

2. Review and approve written procedures for the fabrication and field installation of the UHP piping system.
3. Qualify personnel to work on the UHP piping system, including welder qualifications.
4. Approve weld coupons prior to actual production welding each day.
5. Observe and inspect actual construction work to ensure that agreed-upon procedures are being followed.
6. Witness leak tests on the UHP piping system.
7. Conduct or witness analytical construction verification testing of the gas system for moisture, oxygen, hydrocarbons, and particulates during construction.

To ensure that minimal contamination migrates into the UHP gas supply system during operation, the gas supply piping system must be as leak-tight as possible. Extensive helium leak testing using either a helium-argon test mixture under pressure on the inside of the piping (outboard test) or pure helium on the outside with a vacuum on the inside of the piping (inboard test) must be conducted in which each component, piping system weld, and mechanical joint is separately tested. Specification of leak rates of less than 1.27×10^{-6} ft^3/hr (10^{-9}/sec) at one atmosphere of helium are typically required. Use of either the inboard or outboard test method may be acceptable; each has specific benefits and problems. The designer must take into account which method is to be used.

Construction verification testing of the system may be required at several points in the project installation schedule in addition to certification at completion. The quality assurance representative should also oversee the final analysis of the UHP system to verify that it meets all product purity specifications.

CHECKOUT AND START-UP

The same care that was taken in the design and installation of the UHP gas system must be extended to system start-up. If proper precautions are not taken, all the benefits of a UHP installation can be nullified with the introduction of gross contamination into the system. Typically, the gas supplier oversees the start-up of the gas supply and distribution systems in coordination with the customer. Great care must be exercised so as not to contaminate the system.

The entire system must be systematically purged of the weld argon and test gases and replaced with product gas. For example, if an outboard helium leak test was conducted, it must be recognized that the helium has permeated into the elastomeric materials such as valve seats, and that trapped gases may be downstream of isolation valves that were shut off to isolate segments of the system during testing.

Finally, since the output yield of semiconductor fabs is so dependent upon the purity and cleanliness level of the gas supplies, knowledge of system operation and cleanliness level is necessary at all times to warn of poor operation, back-contamination, or impending performance degradation. Continuous, automatic, computer-controlled, analytical monitors have been developed to very rapidly survey all critical operating parameters, gas purities, and particulate cleanliness. These monitors analyze system maladies and either send out alarms with suggested corrections or correct system adjustments automatically.