

New Technology Is The Next Step In Vacuum Carburizing

Ralph Poor and Stephen Verhoff, Surface Combustion Inc., Maumee, Ohio

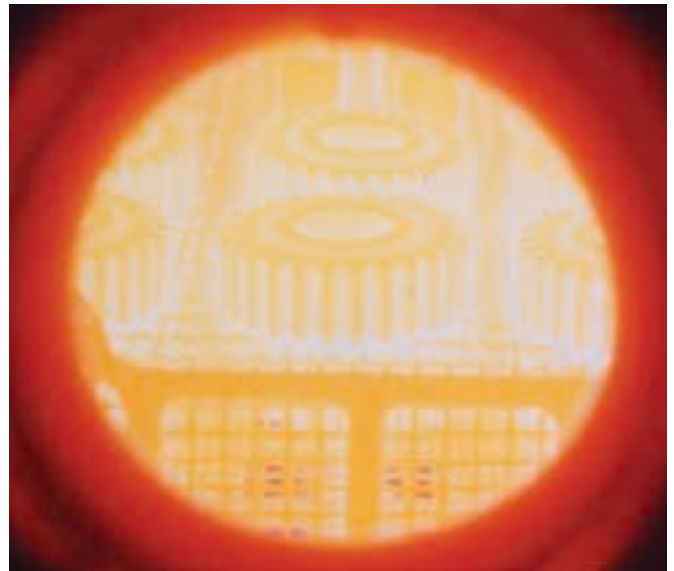
A new vacuum carburizing technology uses a high-purity hydrocarbon compound and a precision controlled liquid injection delivery system to yield repeatable, high-quality results over a wide range of process variables.

A new vacuum carburizing technology, which will be commercially known as VringCarb, has been introduced by Surface Combustion Inc. The process is performed at low pressure in a vacuum furnace using a high-purity naphthene hydrocarbon as the carburizing compound. The furnace is designed to be inert with respect to the naphthene so cracking of the gas occurs only at the workpiece, minimizing carbon deposits. Naphthene is supplied in liquid form to fuel injectors that convert the carburizing medium to a vapor, producing a vapor dispersion around the work, which results in uniform carburizing. Patent applications for the technology were filed in July 2002. Background material on the chemistry involved, purity, delivery, control, process results and furnace design and configurations are presented in this article. Future articles will explore further process results and expand on the tremendous potential of this new processing approach and technology.

Chemical background

In the past, vacuum carburizing process gases largely have been selected from a group of conventional saturated aliphatic hydrocarbons such as methane and propane. More recent developments involve the use of unsaturated aliphatic hydrocarbons. Both of these approaches present issues that continue to challenge those wanting to realize the full benefits of vacuum carburizing.

Methane was a first and logical choice for vacuum carburizing due to its availability and widespread use in conventional atmosphere gas carburizing. Methane is reactive and controllable when used with CO, CO₂, hydrogen, water vapor and other atmosphere constituents commonly found in endothermic atmospheres. However, methane alone is extremely stable, even at elevated temperatures. Therefore, to carburize using methane requires operating the vacuum furnace at about 250 to 400 torr, relatively high pressures (low vacuum levels) with respect to vacuum processing. Operation at these higher



Gears being vacuum carburized

pressures will carburize a workload, but a large amount of carbon drop out or sooting occurs.

The problems associated with using methane have been overcome by using an ion process. Ionization of the methane atmosphere with a high-voltage power supply (about 1000 VDC) allows operation at low pressures (high vacuum levels) on the order of 10 torr. Ionization of methane gas creates a close reaction of the gas with the workpiece without soot drop

out. This process is marketed as ion carburizing.

Propane also has been used as a vacuum carburizing gas with favorable results. Propane is a three-carbon molecular string (C₃H₈) that breaks apart thermally and has a greater tendency to find the workpiece before dropping out as soot in the chamber. Pressures in the 20 to 30 torr range are common, and soot drop out operating at these pressures is greatly reduced over the higher pressure methane approach.



Fig 1 Typical surface quality on vacuum carburized gear



Fig 2 Liquid-hydrocarbon injector mounted to a standard K-25 inlet. Also shown are a simple two-wire 12 VDC electrical connection and a 1/4-in. stainless steel-reinforced supply hose.



Fig 3 Lab vacuum furnaces used for carburizing testing

Due to the inherent limitations of these saturated gases, recent developments have centered around the use of unsaturated aliphatic hydrocarbons such as acetylene and ethylene. Unsaturated hydrocarbons also have a molecular string arrangement, but the bonds are weak and the hydrocarbons are highly reactive even at room temperature. This high level of reactivity makes it easier to carburize. However, a high level of instability comes with this high level of reactivity, which can result in high levels of soot generation. To combat the potential for soot generation, the process is operated at very low pressures.

Unsaturated hydrocarbons also have the ability to join together to form heavier compounds during cooling, which can result in the accumulation of carbon compounds in water-cooled areas within a vacuum furnace.

To overcome these limitations, Surface began an extensive effort to research and evaluate other compounds for suitability in vacuum. The objective was to find a carburizing hydrocarbon that would provide a high level of carburizing potential, coupled with thermal stability, resistance to carbon drop out, and resistance to the formation of undesirable carbon by-products.

The solution was found with the selection of naphthene hydrocarbons. This selection, firmly grounded in existing and known chemical theory, is based on cyclic hydrocarbons. In particular, those hydrocarbons with a saturated 5- and 6-ring structure. This group includes cyclohexane and cyclopentane (see Sidebar). These compounds typically are available in liquid form and are not a gas at room temperature. The naphthene hydrocarbons offer high thermal stability as a result of their ring molecular structure. This thermal stability provides resistance to carbon drop out from thermal

decay. The naphthene hydrocarbons tend to resist thermal breakdown until coming in contact with the workpiece. It is known that naphthene catalysts include platinum, rare earth metals, nickel and iron. This creates a solution for use of naphthene hydrocarbons for carburizing due to the fact that iron is present in all loads where carburizing is desired. The workpiece essentially becomes the catalyst.

Numerous tests using cyclohexane have repeatedly produced clean workpieces (Fig. 1), uniform carburized case depths and little or no carbon deposits, or soot, in the furnace chamber. This

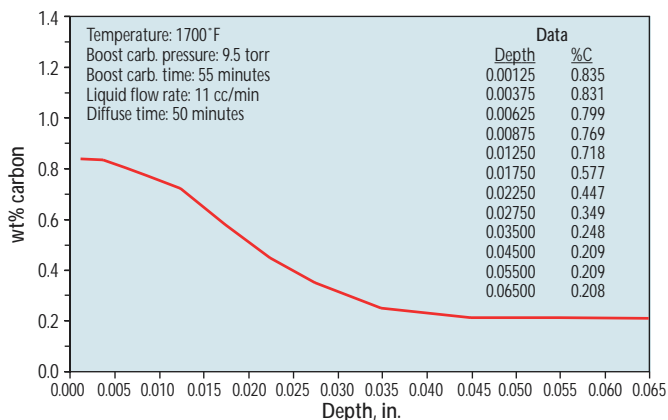


Fig 4 Results of carbon bar analysis for an AISI 8620 bar carburized at 1700°F

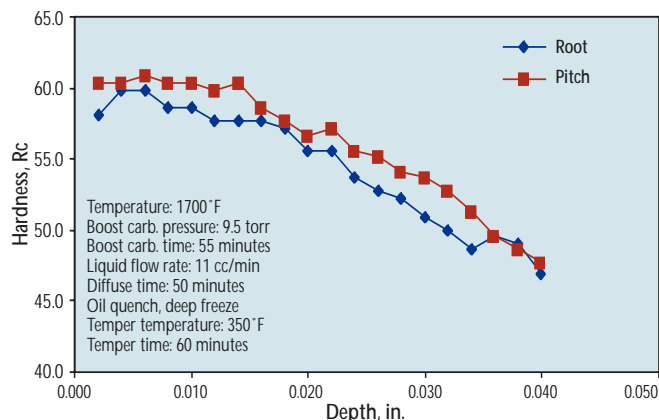


Fig 5 Root to pitch hardness for vacuum carburized AISI 5130 transmission gear

carburizing process works using only naphthene hydrocarbon in the furnace, and the technology does not necessarily require the introduction of any additional gases for carburizing to occur.

Purity, delivery and control

Naphthene hydrocarbons are supplied in liquid form. Standard commercial grade cyclohexane comes in a very pure form of about 99.9% with the other 0.1% "contamination" being other hydrocarbons, which also are suitable for vacuum carburizing. By comparison, many available grades of unsaturated aliphatics have purity in the range of 99.6%, and may contain other constituents such as water vapor, air and traces of CO₂, all of which are considered undesirable in the vacuum process. This means that the high purity of the cyclohexane helps ensure obtaining reproducible, controllable results.

The delivery system for this liquid is a key part of the technology. Surface selected the latest automotive performance fuel injector as the foundation for the liquid delivery system (Fig. 2). The injector is compatible with the liquid naphthene hydrocarbons (present in most gasoline), and is an extremely precise, low cost and highly reliable component.

The injector is connected via a stainless steel reinforced flexible hose to a 15-gal (57 liter) liquid storage tank. The tank is blanketed in argon or nitrogen as the method of providing pressure to the injector(s). The injector is well suited for the

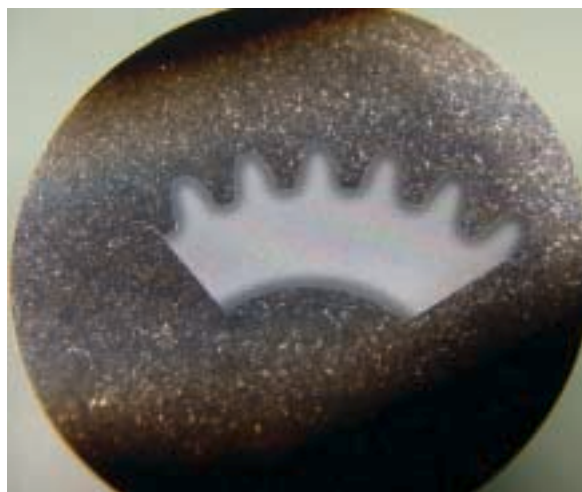


Fig 6 Oil-quenched gear mount



Fig 7 Oil-quenched gear microstructure; 500x

vacuum carburizing application. Ambient temperature and inlet liquid pressures are lower, and adverse conditions such as vibration have been eliminated as compared to automotive applications.

Duty-cycle control of the injector is a real advantage to the device. Injectors can resolve incredibly small changes in millisecond outputs from a computer or controller. The injectors respond to "on-times" in the 2 to 140 ms range, which is the on-time range in an automotive application for each intake stroke. Demonstrated reliability and extremely precise capability to resolve small, subtle changes in timing requests make the injectors an ideal device to deliver a controllable liquid hydrocarbon supply.

The injector system provides precise metering of the carburizing compound into the furnace. Upon entering the furnace, the hydrocarbons are vaporized and break down as they contact the workload. The vapor pressure for naphthenes is in

the range of 80 to 170 torr, so running the furnace significantly below this vapor point ensures the liquid instantly vaporizes and remains a vapor. After the naphthene interacts with the work, the ring is broken and intermediate excess carbon tends to form primarily as methane. As the process progresses and the carbon in the workpiece nears saturation, the level of methane in the furnace tends to increase. Based on the gas chemistry discussed earlier, the excess methane remains stable at temperature in a high vacuum (low pressure) environment.

Test results

Numerous tests at different temperatures and pressures produced the anticipated carburizing results. Oil-quench test results were obtained on a 1-7/16 in. OD by 11/16 in. ID by about 3/4 in. high (36 by 17 by 19 mm) AISI 5130 alloy steel transmission helical gear.

Test gears were loaded in a gas-fired, hot-wall vacuum

furnace (Fig. 3). The furnace chamber and work were heated to a temperature of 1700°F (930°C) under hard vacuum and allowed to soak at temperature for one hour. The boost carburizing segment time was 55 minutes at a pressure of 9.5 torr and a liquid flow rate of 11 cc/min. The diffuse segment time was 50 minutes at a hard vacuum. The parts were oil quenched in vacuum with a production load. Following the quench, the parts were treated by deep freezing and tempered at 350°F (180°C) for one hour. An AISI 8620 carbon bar processed with the load was annealed and analyzed using a Leco carbon determinator (Fig. 4).

Microindentation hardness measurements were taken on polished test gear cross sections to determine hardness profiles (Fig. 5). Transmission helical gears of this type have a complex geometry, and obtaining uniform carburizing results at the pitch and root diameters of the gear is generally considered a primary objective. Figure 5 plots the

hardness and case depth for the carburized gear at its pitch diameter and at its root diameter. The uniformity between pitch and root diameter is considered very tight for any form of carburizing process. As a point of reference, on relatively fine-pitch gears, a root to pitch case depth ratio of 65% typically is obtained using atmosphere gas carburizing. Figure 5 shows a case depth ratio of 90%, which is similar to that achieved using ion carburizing.

Figure 6 shows a mount for the gear processed in this

test. The mount shows the uniformity of the carbon case around the gear tooth and the root-to-pitch uniformity

illustrated in Fig. 5. A micrograph of the same gear shows a highly refined grain structure (Fig. 7).

A second test cycle was performed on a similar AISI 5130 automotive transmission gear. In this test, the

Table 1 AISI 8620 sample carbon bar test results

Temperature, °F (°C)	Time, h	Volume liq. HC (C ₆ H ₁₂), cc	HC (vapor) flow, liter/min	Effective case depth, in. (mm)	Total case depth, in. (mm)	Surface carbon content, %
1600 (870)	3	1942	2.5 (0.05)	0.022 (0.5)	0.032 (0.8)	1.04
1600 (870)	3	1920	2.5	0.022 (0.5)	0.033 (0.84)	1.06
1600 (870)	3	2000	2.5	0.022 (0.5)	0.033 (0.8)	1.06
1700 (930)	2-1/4	1435	2.5	0.03 (0.76)	0.041 (1.04)	1.32
1700 (930)	2-1/4	1460	2.5	0.028 (0.71)	0.04 (1.02)	1.31
1700 (930)	2-1/4	1430	2.5	0.031 (0.79)	0.042 (1.07)	1.32
1800 (980)	1-1/4	800	2.5	0.034 (0.86)	0.045 (1.14)	1.35
1800 (980)	1-1/4	770	2.5	0.036 (0.9)	0.048 (1.22)	1.31
1800 (980)	1-1/4	800	2.5	0.035 (0.89)	0.047 (1.19)	1.30

Understanding cyclohexane

Cyclohexane is a colorless liquid with an annual production of about 400-500 million gallons, primarily for use in the production of nylon, but also used as a solvent, laboratory standard and occasionally to make pesticides and plasticizers. Due to its good combustible properties, it is used as a camp-stove fuel and as an additive in gasoline.

Cyclohexane is available from industrial chemical suppliers in purity levels of 99.9%, and even higher

grades are available from laboratory supply houses. In industrial vacuum carburizing applications, the standard 99.9% commercial grade is adequate for high-quality processing.

Structurally, cyclohexane is a cyclic alkane, technically referred to as a cycloalkane and commonly classified as a naphene in the petrochemical industry. Other naphenes include methylcyclohexane, methylcyclopentane, and cyclopentane. The cyclic structure of cyclohexane is

that of a six-sided ring and is saturated with hydrogen. Saturated hydrocarbons are relatively inert at room temperature and are unaffected

by most acids, alkalis, and oxidizing and reducing agents. The physical properties of cyclohexane are given in the following table.

Environmental and safety concerns

Issue/furnace gas	Cyclohexane	Methanol	Ammonia
NFPA flammability	3	3	1
NFPA health	1	1	3
NFPA reactivity	0	0	0
OSHA exposure limits, ppm TWA	300	200	50
NIOSH exposure limits, ppm TWA	300	200	25
IDLH, ppm	1,300 (10% LEL)	6,000 (10% LEL)	300
Transportation	Class IB flammable liquid	Class IB flammable liquid	
SARA 313 reporting	1,000 lb	5,000 lb	100 lb

TWA = time weighted average over 8-hour day. LEL = lower explosive limit

Chemical Abstract Registry #	110-82-7
Chemical formula	C ₆ H ₁₂
Structural formula	Saturated ring
Atomic weight	84.16
Freezing point, °F (°C)	44 (6.5)
Boiling point @ 1 atm, °F (°C)	177 (80.7)
Vapor pressure @ 100°F, torr (mbar)	168 (224)
Available energy, Btu/gal (kcal/liter)	130,880 (8,713)
Lower flammability limit, %	1.3
Upper flammability limit, %	7.8
Specific gravity @ 60°F, g/cm ³	0.7834
Heat of vaporization @ 1 atm, Btu/lb (cal/g)	154 (85.4)
Auto-ignition temperature, °F (°C)	500 (260)



Ring structure of cyclohexane

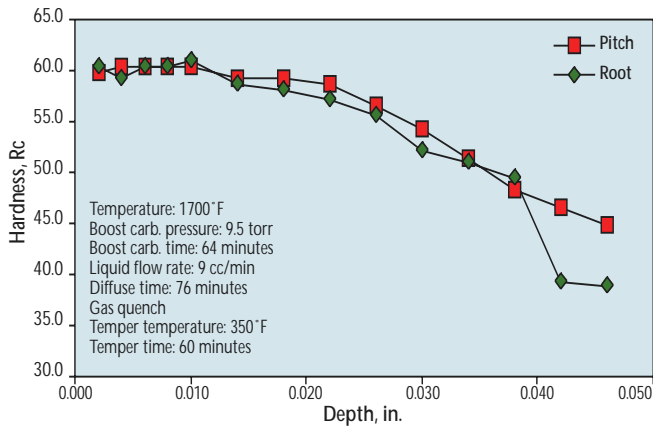


Fig 8 Root to pitch hardness for gas pressure quenched AISI 5130 automotive transmission gear

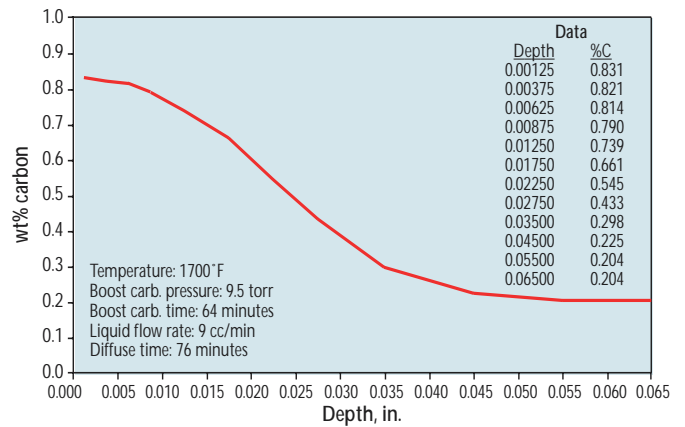


Fig 9 Results of carbon bar analysis for an AISI 8620 bar carburized at 1700°F

gear together with a light load was gas pressure quenched. Root to pitch hardness results are similar to those for the oil-

quenched gear (Fig. 8). The AISI 8620 carbon bar processed with this load also was annealed and analyzed using the Leco carbon deter-

minator (Fig. 9). Figure 10 shows a mount of the gas-quenched gear, illustrating the uniformity of the carbon case around the gear teeth

and the same root to pitch uniformity as the prior test result. A micrograph of the gear again reveals a superior grain structure (Fig. 11).

In addition to the above gear test results, a number of tests were run using a standard sample 8620 test bar at various temperatures, pressures and load dynamics. Data from a sample of these tests are presented in Table 1. Results show that variations in furnace conditions do not impact the ability to achieve high surface carbon and uniform case depths (both effective and total). These data confirm the ability of the process to provide consistent, repeatable results while operating at near saturation throughout a wide range of process parameters using cyclic naphthene hydrocarbon gas.

Furnace design and configurations

In the discussion of chemistry, it was mentioned that iron acts as a catalyst for the cracking reaction of the naphthene hydrocarbon gases. Therefore, you would expect that the desired vacuum furnace design should be "inert" or "transparent" with respect to its ability to cause a catalytic reaction with the cyclic hydrocarbon gases. One of the furnaces used for testing had a conventional graphite board insulation with a graphite foil cover and graphite heating elements. Throughout the testing, the cyclic naphthene hydrocarbons did not react with the graphite. No carbon dropout was

observed on the graphite board or graphite elements. At the end of a carburizing cycle, graphite foil boards were wiped with a wet white paper towel and showed virtually no carbon pick-up. In addition to the conventionally designed dual-wall water-cooled vacuum furnace, extensive testing was performed in a high-temperature hot-wall vacuum furnace heated with four single-end, gas-fired radiant tubes and capable of operating at carburizing temperatures to 1950°F (1065°C). A wide variety of potential materials of construction including ceramic radiant tubes were tested in the furnace with almost no residual carbon left from processing.

Equipment configuration also is an important consideration to achieve repeatable, consistent processing results. It is possible to perform this process in a conventional single chamber vacuum furnace having high pressure quenching capability. Such a configuration is likely to provide a consistent process result load to load. However, such designs have productivity limitations and suffer from the inherent inefficiency of heating and cooling the same chamber repeatedly. Therefore, it is considered likely that the most efficient and productive design configuration will utilize multiple chambers with dedicated heating and cool-

Page 47

OF THE OCTOBER 2002 ISSUE OF
INDUSTRIAL HEATING
CONTAINS A HALF PAGE AD



Fig 10 Gas-quenched gear mount



Fig 11 Gas-quenched gear microstructure; 500X

ing. However, when evaluating multichamber configurations, it is important to consider the requirement for

consistent processing load to load. To achieve this, the conditions for heating, transfer time and quenching

must be the same for each load. Systems must be able to convey the load from heating to quenching in the minimum and exact same time, load to load.

Surface has been a leader in the field of carburizing for over 70 years and has installed over 45 multichamber vacuum and 55 ion processing furnaces with major companies worldwide. Based on our experience and understanding of this process, we now believe that this new technology can be applied to vacuum with results that will dramatically improve the

potential benefits and expand the use of the vacuum carburizing process. Equipment configurations exist that are more efficient, provide high productivity, and the process can now be performed in a clean and controllable manner. **IH**

For more information:
 Ralph P. Poor is Director, Product Development, Surface Combustion Inc., 1700 Indian Wood Circle, PO Box 428, Maumee, OH 43537 USA; tel: 419-891-7150; 800-537-8980; fax: 419-891-7151; e-mail: info@surfacecombustion.com

Vacuum-furnace basics

Atmospheric pressure, the air around us, is 14.7 lb/in.² absolute, or 0 lb/in.² gage. This level of pressure in the world of vacuum is 760 torr, or 760 mm of mercury. As air is removed from a vacuum furnace via a vacuum pump, 760 torr pressure decreases toward 0 torr. From the examples in the article, 10 torr is a point where 99% of atmospheric pressure is removed from the furnace. Actually, 99.993% of the atmosphere is removed before the load is heated, and then the pressure is raised to 10 torr for carburizing.

When the pressure in the furnace drops below 1 torr, the units of measure typically switch from torr to microns. One torr equals 1,000 microns. Typically, the furnace is pumped down to 25 to 50 microns before heat is applied. Therefore, at a pressure of 50 microns, only 0.006% of the atmosphere

still exists in the furnace. At this point, the furnace is heated and pumping continues to remove any contaminants that leave the work surface, such as wash water solution, cutting oils, etc.

Other units of measure for vacuum (pressure levels) include bar, g/cm², and atmospheres. Table A shows equivalent pressures and Table B provides conversion factors to convert from one unit of measure to another.

Table A Pressure equivalents

Atmo-spheres	Psi	Inches water column	Inches Hg	Millimeters Hg	Torr	Microns
1.07	15.7	435	32	812		
1.03	15.2	421	31	786		
1.00	14.7	407	30	760	760	760,000.0
0.5	7.35	203.5	15	380	380	380,000.0
0.068	1.0	27.7	2.03	51.7	51.7	51,000.0
	0.49	13.6	1.0	25.4	25.4	25,468.0
	0.036	1.0	0.074	1.87	1.87	1,870.0
	0.019	0.54	0.039	1.0	1.0	1,000.0
				0.1	1 × 10 ⁻¹	100.0
				0.01	1 × 10 ⁻²	10.0
				0.001	1 × 10 ⁻³	1.0
				0.0001	1 × 10 ⁻⁴	0.1
					1 × 10 ⁻⁵	0.01
					1 × 10 ⁻⁶	0.001

Table B Pressure-conversion factors

To convert from ↓	To these units, multiply by				
	Atmosphere	Psi (lb/in ²)	Inches (Hg)	Torr (mm Hg)	Microns
Atmosphere		14.696	29.92	760.0	760,000
Bar	0.987	14.5	29.51	750.0	750,000
Kg/cm ²	0.968	14.22	28.96	736.0	736,000
Psi (lb/in ²)	0.068		2.036	51.5	51,500
Inches (Hg)	0.0334	0.491		25.4	25,400
Feet (H ₂ O)	0.0295	0.434	0.8826	22.2	22,200
Inches (H ₂ O)	0.00246	0.0361	0.0736	1.85	1,850
Torr (mm Hg)	0.00132	0.0195	0.0394		1,000
Millibar	0.000987	0.0145	0.0295	0.750	750
G/cm ²	0.000968	0.0142	0.0290	0.725	725
Microns	1.32 × 10 ⁻⁶	1.95 × 10 ⁻⁵	3.94 × 10 ⁻⁵	10 ³	