KNOWLEDGE

The Nature of Hydrogen Gas and the Benefits of Coriolis Fluid Measurement Technology

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Hydrogen is considered a difficult industrial gas to measure. Due to its low molecular weight and therefore low operating density, traditional technologies such as differential pressure, vortex, or thermal mass experience difficulties measuring hydrogen flow. Since 1992, developments in Coriolis technology have resulted in a breakthrough in hydrogen metering. Improvements in both sensitivity to flow and immunity to flow-induced noise have resulted in highly accurate hydroge metering capability over wide flow ranges and operating conditions.

This paper presents the benefits of using Coriolis technology for metering hydrogen, and includes several applications from the chemical, refining, and food industries. An overview of hydrogen is also presented, including the nature of the gas, area classifications and approvals, plus a discussion of commonly accepted piping practices.



Measurement of hydrogen for production of fatty acids

Key benefits of Coriolis-based measurement of hydrogen

- Higher accuracy due to Coriolis measurement, over wide turndown. Independent of changes in pressure and temperature. Uncertainties of volumetric measurement and conversion to standard conditions (inferred mass) eliminated. Molecular weight changes do not affect metering accuracy. Third-party testing at 100:1 turndown and 60:1 operating density range confirms accuracy and repeatability on gas flows.
- Lower routine maintenance costs because Coriolis meters have no moving parts. Calibration factor does not shift over

time, eliminating need for routine proving. Mass calibration factor applies to any gas. Gas meters can be proved using liquid medium and traditional proving devices.

• Easier to install and maintain. No straight pipe runs or filters/strainers required. Insensitive to swirl effects. Sensor can be installed in horizontal or vertical pipe runs.

Hydrogen overview

Hydrogen is a valuable and widely metered industrial gas with numerous important applications. Hydrogen is produced and/or purified using catalytic or steam reforming of hydrocarbon mixtures, electrolysis of water, and catalytic reforming in petroleum refineries. It is often generated on-site, or transferred from high pressure cylinders provided by major industrial gas suppliers.

Hydrogen is highly reactive in the presence of certain catalysts and can dramatically improve the commercial value of materials, such as edible oils. Hydrogen is considered difficult to measure because of its small molecular size, low molecular weight and therefore low operating density. Accurate, reliable flow metering is important to assure final product quality or yield, as well as plant safety.

Because hydrogen is very explosive, it must be handled carefully. The minimum ignition energy of hydrogen is an order of magnitude smaller than that of hydrocarbons. To ensure plant safety, leak points in hydrogen piping systems must be eliminated.

Measuring hydrogen

Hydrogen is considered a difficult gas to meter using traditional volumetric technologies because it has low operating gas density. Micro Motion[®] Coriolis flowmeters are replacing these technologies because mass is measured directly. Coriolis meters are also unaffected by inlet flow conditions (swirl effects), and thus require no straight pipe run or other flow conditioning.

Coriolis meters require virtually no maintenance. Because mass is measured directly, there is no need for pressure and temperature compensation to correct back to standard conditions. Although mass is being measured, transmitter outputs can be configured in familiar units, such as scfm (standard cubic feet per minute) or NM^3/h (normal cubic meters per hour).

This paper describes a variety of ways in which the Micro Motion Coriolis mass flowmeter is being used to improve hydrogen gas measurement.





Nature of the gas

Hydrogen gas (chemical formula H_2) is composed of two hydrogen atoms, and is colorless, odorless, tasteless, nontoxic, and flammable. With a molecular weight of 2 and a specific gravity of 0.0695, hydrogen is considered the lightest gas – over 14 times lighter than air (mw=28.8).

Hydrogen is extremely flammable. At atmospheric pressure, a mixture ranging from 4.0 to 74.0% is flammable. Since hydrogen has a very low minimum ignition energy (0.02 mJ – an order of magnitude lower than hydrocarbon gases), significant precautions must be taken to prevent leaks. A simple spark resulting from sources such as static electricity buildup can ignite hydrogen.



Special Considerations

<u>Area Classification and Approvals</u>: Hydrogen is considered a Group B gas, per the National Electric Code (NEC). Other Group B gases include acrolein (C_3H_4O), arsine (AsH₃), butadiene (C_4H_6), ethylene oxide (EO or C_2H_4O), propylene oxide (PO or C_3H_6O), propylnitrate ($C_3H_7NO_2$), and manufactured gases containing more than 30% hydrogen by volume.

Micro Motion flowmeters are intrinsically safe. Although the ELITE[®] line is not rated for use with Group B gases such as hydrogen in a Class I, Division I area, most Micro Motion meters measuring hydrogen can be installed in a suitable area of the plant to meet safety approvals.

Hydrogen Embrittlement: Due to its small molecular size, hydrogen can diffuse into the interstitial spaces in the crystalline lattice of many metals, which may cause a loss of tensile ductility (strength). This effect is called hydrogen embrittlement and primarily affects high strength steels other than the stainless steels. Austenitic stainless steels, such as 304L and 316L are virtually immune to hydrogen embrittlement, as is the Ni-Cr-Mo structure of Hastelloy C-22. The use of these materials in Micro Motion Coriolis meters assures long, reliable life in hydrogen service.

Piping precautions

<u>Process Connections</u>: Hydrogen may leak out of a system that is gas tight with respect to air or other common gases at equivalent pressure. The Compressed Gas Association (CGA) guideline CGA G-5.4-1992 states: 5.1.2 Joints in piping and tubing shall preferably be welded for leak tightness and fire safety. Joints may also be threaded, flanged, brazed or made with a suitable mechanical fitting. Soft solder joints are not permitted.

5.1.3 Threaded joints are acceptable on hydrogen piping but should be minimized to reduce potential for leakage.

5.1.4 Flanged joints on hydrogen piping shall have a metal-tometal seal, or a gasket of material such as graphite, which is difficult to ignite, or be suitably protected from a fire safety standpoint.

Micro Motion sensors utilize an all-welded design. Because pressure taps and temperature probes are not required to correct the flow reading back to volumetric reference conditions, system piping joints are minimized. Sensors are available in flanged and compression type Cajon™ or Swagelok™ fittings.

Detailed specifications covering hydrogen piping, manifold systems, and bulk supply systems are presented in NFPA 50A, *Standard for Gaseous Hydrogen Systems at Consumer Sites*. Liquid hydrogen systems are covered under NFPA 50B.

Applications

Hydrogen gas is used in numerous processes, including primary metal production, manufacture of rocket propellant, fuel cells, metal hydrides, semiconductors, heat treating metals, production of ammonia, hydrogenation of edible oils, and liquid petroleum fractions. Hydrogen gas is also used in the production of numerous chemicals, including the production of fatty alcohols from their corresponding acids or aldehydes. Large electrical generators are often run with an internal hydrogen atmosphere to reduce windage losses (internal wind resistance caused by the spinning rotor) and remove heat. Fiscal metering or custody transfer (CT) is an important aspect of hydrogen flowmetering, due to the high relative value of hydrogen gas.



Hydrogen cost-accounting

Hydrogen is a commonly used gas in research and development, and is typically measured at very low flow rates.

Hydrogen cost-accounting

In this lube oil refinery application, a Micro Motion Coriolis flowmeter measures makeup hydrogen to the hydrotreater. Traditional dP/orifice technology was previously used, and was the basis for billing. Dramatic cost savings were generated from the switch to Coriolis, confirming the refiner's suspicion that they were being charged for delivery system losses.

Now, the refinery only pays for the hydrogen it actually uses, and is not billed for the gas lost in the transfer system. Switching to Micro Motion Coriolis technology has produced annual savings of \$150,000 USD, a payback in less than one month.

Custody transfer of hydrogen

This ELITE model CMF025 meter is installed as the "cash register" between a major midwestern USA industrial gas supplier and a chemical plant. Hydrogen is supplied continuously at flow rates of up to 325 scfm=1.7 lb/min (550 NM³/h = 46 kg/h), and charges a bank of cylinders at the chemical plant. These cylinders then handle the peak demand of the chemical plant. See Figure 1.



Figure 1. Custody transfer to chemical plant; ELITE CMF025H

The Micro Motion Coriolis meter was chosen over a traditional orifice plate/dP setup. As a result, the requirement of measuring temperature and pressure for compensation back to standard conditions, and the necessity of a flow computer have been eliminated. Also, routine maintenance, such as recalibration of the pressure and temperature transmitters, plus concerns over the orifice meter installation itself, have been avoided.

According to the customer, the Micro Motion meter was a perfect fit for his application:

"The (small) size of the hydrogen molecule is the issue, and the low gas density makes measurement with traditional volumetric meters tough. We were looking for rangeability, and simplicity... The Micro Motion meter is a very simple device that is easy for us to use. We've never had to recalibrate it."

– Regional Sales Engineer, major industrial gas supplier, Midwestern U.S.



ELITE CMF025-Hastelloy measures hydrogen for custody transfer

Fatty acid and fatty alcohol production

Hydrogen is used as a reactor feedstock in the continuous production of fatty acids and alcohols. Fatty acids are used mainly in detergents, soaps, cosmetics, paper, and textiles. The bulk of fatty alcohol production is used in the production of surfactants. High pressure hydrolysis converts fats to fatty acids. Fatty acids can be further converted to fatty alcohols by hydrogenation. Accurate control of this hydrogen feed is critical to assuring product quality. Measuring hydrogen consumption is also common.

In this application, a manufacturer of fatty acids experienced continuous problems with an orifice/dP flowmeter. Despite full pressure and temperature compensation on the dP meter, final product composition varied significantly, creating suspicion that the flowmeter was highly inaccurate. This plant also produces hydrogen in another unit. More hydrogen is produced than is consumed in the fatty alcohol unit; therefore, a surplus should exist. Instead, makeup hydrogen was required, exposing a serious measurement problem and big expense for the plant – since makeup hydrogen had to be purchased. See Figure 2.

This particular customer remarked, "We were always using more (hydrogen) than we were making. We could never get an apples to apples comparison. This went on for years."

The customer added Micro Motion ELITE meters for three purposes: rate control to the fatty acid reactors, inventory control to the plant, and inventory control for hydrogen produced.



Figure 2. Reactor feed for fatty acid/alcohol production

After installing the 1" (25mm) ELITE CMF100 flowmeter, fatty acid quality was dramatically improved, and the hydrogen mass balance closed for the first time ever.

The ELITE CMF100 replaced the orifice plate/flow computer combination. This dramatically improved system accuracy, made the system easier to mass balance, and improved product quality.

"Micro Motion told us about the ELITE meters on gas ... Now, we can compare apples to apples. And now, we can check our (material) balance as well as measure hydrogen to storage."

- Chemical manufacturer, Southeastern U.S.

Manufacture of edible oils

In this application, Micro Motion meters are used in lieu of vortex technology to add precise amounts of hydrogen gas to selectively hydrogenate edible oils.

The vortex meters previously used were not sufficiently accurate to meter the wide flow range necessary to produce a top quality final product.

Edible fats and oils are susceptible to oxidation and rancidity. Thus, they are selectively hydrogenated to extend shelf life for processed foods, and establish target melting points for ease of processing with automated equipment.

The greater the degree of unsaturation, the softer the fat, and lower its melting point. Complete saturation converts liquid oil into solid shortening. Partial hydrogenation results in an intermediate degree of saturation and solidification. Soybean and Canola are the most frequently used oils.

Hydrogenation to saturate fatty acid double bonds is carried out by whipping deaerated hot oil with hydrogen gas, plus a nickel catalyst in a closed reactor vessel (converter). When the desired reaction is complete, any excess hydrogen is removed by vacuum, and the catalyst is recovered by filtration. Hydrogen is fed based on reaction stoichiometry. Since the reaction is generally exothermic (heat generating), accurate rate control is critical for both safety as well as product quality.



ELITE CMF300: Hyperpure product production

To prevent over-hydrogenation, slightly less hydrogen is charged to the batch reactor than the reaction chemistry (recipe) calls for.

The oil is ruined if "overshoot" occurs. After the hydrogen fed to the (batch) reaction is consumed, a sample of the oil is taken to the lab to determine the extent of hydrogenation. The batch is then "fine-tuned" by adding additional small amounts of hydrogen to achieve the target product specification.

Since one hydrogen flowmeter is used to handle both the main charge as well as the subsequent small amounts of hydrogen, the meter's ability to accurately handle a wide range of flows is critical. Thermal mass meters are sometimes used in this application, but are known to experience drift over time. Since in situ gas proving is difficult, reliable, drift-free Coriolis technology from Micro Motion eliminates these concerns.

Vortex meters are also sometimes used, but the 50:1 required turndown for this application exceeds their capabilities.

Final edible oil is then cooled and accurately packaged using the Micro Motion meter. See Figure 3.

Significant improvements in product quality, as well as increases in throughput, have been greatly facilitated by the use of ELITE CMF100 flowmeters in this application.

Fragrance manufacture

Two Micro Motion Model D meters are used for ratio feed control to a gas phase reactor vessel in the production of a key ingredient for men's after-shave and cologne. Quality control is paramount, but was previously very difficult to achieve with manual control and a batch reactor.

Manual charging based on reaction stoichiometry between hydrogen and carbon monoxide (CO) was attempted using pressure gauge readings. The addition of CO and H_2 was inferred by measuring pressure and temperature in the reactor vessel. This method is inherently inaccurate, due to the challenge of manually reading pressure and temperature gauges.

According to the customer:

"It (manual control) never worked, and took a lot of operator involvement to finally get the mixture right."

An upgrade to on-line mass flow measurement using Micro Motion's Coriolis technology enabled conversion of the reaction scheme from batch to continuous, increasing both plant efficiency and throughput. The multiple attempts previously required to obtain the correct gas mixture were eliminated, and an on-spec product was produced on a continuous basis. See Figure 4.

In addition to increased plant profitability, reduced product quality variation allowed Statistical Process Control (SPC) limits to be tightened.

Highly accurate gas flow measurement, and ease of installation due to the lack of straight run requirements make Coriolis



Figure 3. Manufacture of edible oil using an ELITE CMF100

meters ideal for the application. The meters in this installation have been maintenance-free since they were installed more than 5 years ago.

"When you (Micro Motion) asked about the meters, I had to think about where they were because I hadn't needed to touch them for a while...Every experience with Micro Motion has been positive; we're moving more and more toward mass flow."

– Instruments & Controls Supervisor, Flavors and fragrance manufacturer, Northeastern U.S.

Hydrogen for production of cyclohexane

Hydrogen is fed from a hydrogen trailer ("6-pack" at 2300 psi) to an autoclave. Previously, the measurement of "uptake" hydrogen was based on inferred mass from pressure and temperature readings on the autoclave itself.

The previous measurement technology was generally acceptable, but exhibited some problems at the low flow end, especially at 50 psi (3 bar) pressure. The autoclave is a well-agitated, gas phase reaction vessel. See Figure 5.

Numerous and varied hydrogenation reactions are carried out,



Model DS012 and DS040-Stainless Steel at fragrance manufacturer in northeastern U.S.

some selective (site specific) and some common. Examples are benzene to cyclohexane, and various alcohols to ketones. These are batch reactions, involving an initial charge of hydrogen based on reaction chemistry. A slight undercharge is intentionally planned, with sampling and lab analysis performed to fine-tune the batch and achieve the target product yield.

Traditional dP/orifice technology is often used in this type of batch reaction. Due to limited low-flow capability, several "fine-tuning" steps are required, in order to avoid over-hydrogenation.



Figure 4. Production of men's fragrance using Micro Motion Models DS012 and DS040

Because Micro Motion meters are accurate even at high turndowns, the entire flow rate can now be accurately measured.

"Now, with the Micro Motion meters, we can get it right the first time."

Savings in raw materials and significant improvements in simplicity and speed of operation have been accomplished since upgrading to Micro Motion Coriolis meters.

Corrosion prevention

A Coriolis meter from Micro Motion is used to help control corrosion in burners processing corrosive gases. In the production of silicon tetra chloride or SiCl₄ (used for AerosilTM, an inert filler used for ketchup, toothpaste, etc.), a precise amount of

excess hydrogen must be maintained in the burner feed to prevent chlorine corrosion. Accuracy is a key factor in this application.

Previous burner corrosion problems were traced to inadequate performance from the dP and integral orifice meters, resulting in their replacement with Micro Motion ELITE meters. Due to the precise measurement of hydrogen by the Micro Motion meters, corrosion in the burner has been virtually eliminated. As an added benefit, the increased accuracy of the Micro Motion meters have boosted the reaction yield of SiCl₄. See Figure 6.

"Because of the accuracy and reliability of the Micro Motion meter, we have not had any more problems with corrosion in the burner."

- Specialty chemical manufacturer, Southeastern U.S.



Figure 5. Cyclohexane from Benzene using Micro Motion Model DH038

Sizing and selection

Sizing and selection of flowmeters for gaseous hydrogen service is virtually identical to the sizing procedure for other Micro Motion gas applications. Acceptable pressure drop and desired accuracy over the flow range of interest are the two primary criteria. Micro Motion provides a sizing and selection program (www.micromotion.com) that allows a user to enter specific process conditions that drive a sizing and selection recommendation. User-entered variable include flow rate, operating pressure and temperature. The user makes the final selection based on allowable pressure drop and desired accuracy.



Figure 6. Corrosion control, SiCl₄ production using Micro Motion ELITE CMF050s



Cyclohexane production

Micro Motion's ELITE sensors and MVD electronics are strongly preferred for gas service in general, and hydrogen applications in particular. The high sensitivity and stability of the ELITE line of sensors, combined with the latest in digital signal processing capability of the MVD electronics platform provide the superior immunity to flow-induced noise and enhanced low flow capability - the two prerequisites for hydrogen gas measurement applications. With sizes ranging from 1/8" (3mm) ELITE CMF010 to the 4" (100mm) ELITE CMF400, hydrogen flow rates from virtually zero up to approximately 385,000 scfm (2000 lb/m) or 654,120 Nm3/h (54,420 kg/h) can be measured using a single flowmeter¹.



Reaction chemistry

Hydrogen meter sizing typically result in flowmeter (tube) velocities much higher than those seen for heavier gases such as nitrogen and natural gas. Velocities up to and exceeding 1000 ft.sec (320 m/sec) through the flow tubes are not a concern. Meter sizing is based on acceptable pressure drop and flow accuracy. Micro Motion has conducted extensive third-party testing showing excellent results on gas velocities up to Mach 0.5.

Sonic velocity is defined as the speed of sound in a given media (gas). Gas cannot travel faster than its own standing shock wave, regardless of how much the inlet pressure is increased. Sonic nozzles or venturis utilize this physical phenomenon to determine gas velocity through the nozzle,

¹ Process conditions are 1450 psi (100 bar) at ambient temperature.

enabling this transfer standard to be used as a proving or calibration device.

Sonic velocity depends primarily on gas molecular weight and temperature. Whereas sonic velocity of air is around 1025



Corrosion prevention

ft/sec (331 m/sec) at STP (TPN), sonic velocity of hydrogen is much higher, at 3980 ft/sec (1284 m/sec).

Pressure drop and accuracy are the two primary criteria for sizing a Coriolis flowmeter for hydrogen. Gas velocity through the sensor, with very acceptable pressure drop, can often approach 1000 ft/sec (322 m/sec) or more. Since the sonic velocity of hydrogen is so high, this is only 0.25 Mach, much lower than the 0.5 Mach reached in testing. As a result, high gas velocities through the sensor are not a concern from a measurement perspective. If grit or particulate matter is present in the piping system, an upstream strainer should be considered to protect the sensor from possible damage due to erosion.

Why is hydrogen gas so difficult to measure with volumetric technologies?

Gas mass vs. volume

Remember Avogadro's Principle from your chemistry or physics coursework? Count Amedeo Avogadro, a physicist from the early 1900s, discovered that equal volumes at the same temperature and pressure contain an equal number of gas molecules, regardless of the gas. This is why actual volume flow is converted to standard conditions, to determine how much material (molecules) is present.

Mass (moles) is the basis of all gas reactions, blending, and legal transfer. To convert an actual volume flow to standard (normal) conditions, actual volume, plus pressure and temperature, must be measured. Gas composition must be known or measured, compressibility factor (z) determined, and the computation performed. Only then, can inferred mass can be calculated in a flow computer, PLC or DCS.

Multiple sources of error, calculations and less-than-perfect actual volumetric measurement combine to make the standard (normal) volume less completely accurate than it appears. For a light gas such as hydrogen, propagation of these errors can easily result in inaccuracies for inferred hydrogen mass of up to 10%.

Coriolis measurement of H_2 eliminates these sources of error and typically offers accuracies better than 0.35% (when using ELITE with MVD) of actual mass. Coriolis technology also eliminates straight pipe runs and other flow conditioning requirements. Measuring mass directly is simple, and transmitter outputs can easily be configured in standard or normal volume units such as scfm or NM³/h.

Coriolis accuracy in familiar units:

 $Q_{v @ STP} = Q_m/standard density @ STP$ $Q_v (scfm) = Q_m (lb/min)/0.00523 lb/ft^3$ where STP = 68 deg F and 14.7 psia



Avogadro's Principle: Equal gas volumes at the same temperature and pressure contain the same number of gas molecurles.

Summary

Hydrogen is a commonly measured and valuable industrial gas that is used in a wide variety of processes. It is highly flammable, with low ignition energy, so leaks in process systems must be minimized to maximize safety for plant personnel.

Reliable, accurate flow measurement is critical to increasing product quality and yield. Existing volumetric technologies such as dP, turbine, and vortex are limited by accuracy. When using these technologies to measure hydrogen, accuracy performance is usually no better than \pm 3-8%. Turndown is also limited, and turbine meter moving parts jeopardize reliability. Thermal mass meters have often been used, but are somewhat maintenance intensive, tend to drift with ambient temperature swings, and must be calibrated for each specific gas.



More recently, Coriolis meters from Micro Motion have been used in hydrogen service with excellent results. Third-party test data confirms useable turndown in excess of 100:1 flow range, and 60:1 operating density range. Coriolis meters from Micro Motion also eliminate straight run requirements and routine maintenance - dramatically lowering the cost of ownership. Direct mass measurement, synonymous with standard or normal volumetric units, is fundamentally more accurate and eliminates the need for flow correction factors based on pressure and temperature. True mass accuracy of ± 0.5% of actual mass rate ± sensor zero stability make Micro Motion Coriolis meters one of the most accurate gas flowmeters under any condition.

With an installed base of over 400,000 flow and density sensors worldwide, Micro Motion can provide the measurement expertise and support you need. Extensive third-party testing and more than 25,000 gas phase applications around the world have proven the numerous benefits of using Micro Motion Coriolis technology to measure hydrogen gas.





19120 scfm (3020 NM3/h at 68°F (0°C reference) H_2

Gases measured with Micro Motion meters include:
Acetylene (C_2H_2)
Air
Ammonia (NH ₃)
Argon (Ar)
Boron Trifluoride (BF ₃)
Butadiene (C ₄ H ₆)
Butane (C ₄ H ₁₀)
CNG
Carbon Dioxide (CO ₂)
Carbon Monoxide (CO) Chlorine (Cl ₂)
Ethane (C_2H_6)
Ethylene (C_2H_4)
Freon
Fuel gas
Helium (He) Hexape (CoHere)
Hydrogen Chloride (HCI)
Hydrogen Cyanide (HCN)
Hydrogen Sulfide (H ₂ S)
Iso-Butane
Methane (CH ₄)
Methyl Chloride (CH ₃ Cl)
Methyl Mercaptan
Natural Gas Neon (Ne)
Nitrogen (N ₂)
Oxygen (O ₂)
Phosgene (COCI ₂)
Propane (C ₃ H ₈)
Propylene (\tilde{C}_3H_6)
Refrigerant Gases
Steam
TrifluoroEthylene-TFE (C ₂ HF ₃)
Vent gas

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Micro Motion supports PlantWeb field-based architecture, a scalable way to use open and interoperable devices and systems to build process solutions of the future.

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