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Special Documentation
Validation of TDLAS gas analyzers

Endress+Hauser
People for Process Automation

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1 About this document

1.1 Document function

This manual is a special documentation for any Endress+Hauser tunable diode laser absorption spectroscopy (TDLAS) and does not replace the operating instructions included in the scope of supply. It serves as a reference for validation of any tunable diode absorption spectroscopy (TDLAS) product.

1.2 Content and scope

This Special Documentation contains a range of information, including:

Validation methodology
Design options
Ranges of gases
Validation examples

The information and safety instructions in the operating instructions pertaining to the measuring device must always be observed.

1.3 Symbols

1.3.1 Safety symbols

Structure of Information	Meaning
⚠ WARNING **Causes (/consequences)** Consequences of noncompliance (if applicable) ▸ Corrective action	This symbol alerts you to a dangerous situation. Failure to avoid the dangerous situation can result in a fatal or serious injury.
⚠ CAUTION **Causes (/consequences)** Consequences of noncompliance (if applicable) ▸ Corrective action	This symbol alerts you to a dangerous situation. Failure to avoid this situation can result in minor or more serious injuries.
NOTICE **Cause/situation** Consequences of noncompliance (if applicable) ▸ Action/note	This symbol alerts you to situations which may result in damage to property.

1.3.2 Symbols for certain types of information

Symbol	Meaning
✓	Permitted Procedures, processes, or actions that are permitted.
✗	Forbidden Procedures, processes, or actions that are forbidden.
i	Tip Indicates additional information.
	Reference to documentation
	Reference to page
	Reference to graphic
▶	Notice or individual step to be observed
1., 2., 3. ...	Series of steps

Symbol	Meaning
↳	Result of a step

1.3.3 Symbols in graphics

Symbol	Meaning
1, 2, 3 ...	Item numbers
A, B, C, ...	Views
A-A, B-B, C-C, ...	Sections

1.4 Documentation

All documentation is available:

On the media device supplied (not included in the delivery for all device versions)

On the Endress+Hauser Operations App for smartphone

In the Downloads area of the Endress+Hauser website: www.endress.com/downloads

For additional instruction manuals, refer to the Endress+Hauser website to download the published documentation: www.endress.com.

1.5 Registered trademarks

Modbus®
Registered trademark of SCHNEIDER AUTOMATION, INC.

1.5.1 Manufacturer address

Endress+Hauser
11027 Arrow Route
Rancho Cucamonga, CA 91730
United States
www.endress.com

1.6 U.S. export compliance

The policy of Endress+Hauser is in strict compliance with U.S. export control laws as detailed on the website of the Bureau of Industry and Security at the U.S. Department of Commerce.

2 Basic safety instructions

Each analyzer shipped from the factory includes safety instructions and documentation for the responsible party and/or operator of the equipment for the purpose of installation and maintenance.

WARNING

▶ **To service or operate the analyzer, technicians are expected to be trained and follow all safety protocols that have been established by the customer in accordance with the area hazard classification.** This may include, but is not limited to, toxic and flammable gas monitoring protocols, lockout/tagout procedures, the use of personal protective equipment (PPE) requirements, hot work permits, and other precautions that address safety concerns related to the use and operation of process equipment located in hazardous areas.

2.1 Requirements for the personnel

Personnel involved in installation, commissioning, diagnostics, and maintenance of the device must meet the following requirements:

- Trained with relevant qualification for this specific function and task
- Trained in explosion protection
- Trained in national and local regulations and guidelines (e.g., CEC, NEC ATEX/IECEx, or UKEX)
- Trained with lockout/tag-out procedures, toxic gas monitoring protocols and PPE (personal protection equipment) requirements
- Read and understand the instructions in the manual and supplementary documentation as well as the certificates (depending on the application) before starting work
- Follow instructions and comply with basic conditions

WARNING

▶ **Substitution of components is not permitted**. Substitution of components may impair intrinsic safety.

2.2 Intended use

The devices mentioned in this manual are intended for the measurement of various industrial gases, which may be potentially explosive, flammable, toxic, or corrosive.

Measurement devices used in hazardous areas are marked accordingly on the nameplate.

To ensure that the analyzer remains in proper condition during its lifespan:

- Keep within specified pressure and temperature range.
- Only use the analyzer in full compliance with data on the nameplate and general conditions listed in the operating instructions and supplementary documentation.
- Use the analyzer only gases where the process-wetted materials are sufficiently resistant.

2.2.1 Incorrect use

Not using the equipment according to manufacturer instructions can compromise safety. The manufacturer is not liable for damage caused by improper or undesignated operation.

2.3 Workplace safety

Wear the required personal protective equipment as per national regulations when working on the device.

2.4 Operational safety

WARNING

Operate the device only if it is in proper working condition, free from errors and faults.

▶ The operator is responsible for interference-free option of the device.

2.5 Modifications to the device

Unauthorized modifications to the device are not permitted and can lead to unforeseeable dangers. If modifications are required, consult with the manufacturer.

2.5.1 Repair

To ensure continued operational safety and reliability:

- Conduct repairs on the device only if they are expressly permitted by the manufacturer.
- Observe federal/national regulations pertaining to repair of an electrical device.
- Use only original spare parts and accessories.

2.6 Product safety

The TDLAS gas analyzer is designed in accordance with good engineering practice to meet state-of-the-art safety requirements, has been tested, and left the factory in a condition in which it is safe to operate.

It meets general safety standards and legal requirements. It also complies with the EU directives listed in the specific EU Declaration of Conformity. Endress+Hauser confirms this by affixing the CE mark to the analyzer system.

2.7 IT security

Our warranty is valid only if the device is installed and used as described in the operating instructions. The device is equipped with security mechanisms to protect it against any inadvertent changes to the settings.

IT security measures, which provide additional protection for the device and associated data transfer, must be implemented by the operators themselves in line with their security standards.

3 Validation description

One of the benefits of TDLAS gas analyzers is the long-term stability of optical measurement. After factory burn-in and calibration, very minimal drift of the wavelength occurs over the lifespan of the device. Field calibration is not required. The performance of the analyzer can be confirmed through a validation process. Gas with a known concentration of the analyte is passed through the analyzer. Then, the measurement of this validation gas is compared with its stated value to determine if the analyzer performance meets the expectations of the user.

Validation is frequently confused with calibration; however, the terms have different meanings.

- Calibration implies a change to the measurement is applied to analyzer's settings. Readings before and after calibration is different when exposed to the same analyte concentration.
- Validation differs from calibration in that no actual adjustment of the measurement occurs. A validation simply checks the analyzers performance. A proper validation ensures the integrity of the analyzer and gives the user confidence in their measurement.

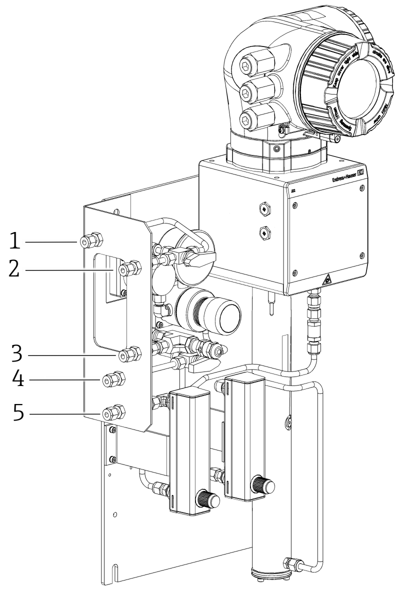


Figure 1: View of J22 connections

#	Name:
1	Sample purge inlet (optional)
2	Sample inlet
3	Relief vent (optional)
4	Reference gas inlet
5	Sample vent

4 Validation methodology

Endress+Hauser Optical Analysis employs two validation methods for our TDLAS gas analyzers, certified validation gas cylinders containing validation gases, and permeation tubes.

4.1 Certified gas cylinders

Certified validation gases are binary gas mixtures that combine the analyte of a known concentration with an inert background gas. The validation gas is supplied by a third-party vendor such as Linde, Air Liquide, or AirGas, and come in high pressure cylinders. The concentration of analyte as well as the gas background can be specified at time of order. Common validation gas backgrounds are listed below:

- **Methane background**: Methane has been historically recommended for the validation of moisture (H_2O) and hydrogen sulfide (H_2S) analyzers used in natural gas applications. In recent times, methane background validation gases have become less popular due to the hazardous nature of the gas as well as environmental concerns about methane as a greenhouse gas.

- **Nitrogen background:** Nitrogen is a common background that may be used in place of methane. Nitrogen has little to no interference at typical TDLAS wavelengths and is non-flammable.

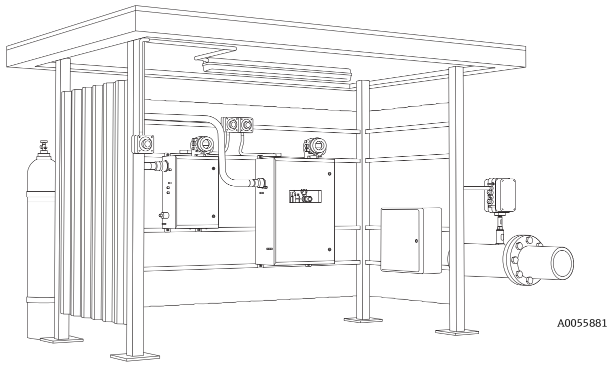


Figure 2: Validation gases are supplied in high pressure gas cylinders and stored on-site near the analyzer installation

- **Carbon dioxide background**: Carbon dioxide (CO_2) validation gas backgrounds are less common. CO_2 may be used in carbon capture applications and is offered as a validation option for our H_2S analyzers. CO_2 backgrounds are nonflammable which adds to their growing popularity; however, chemical compatibility of the analyte with CO^2 must be considered.

4.2 Permeation devices

Trace moisture measurements are often at such low concentrations that commercial validation gases are not available. These trace analyzers use a permeation device (often referred to as a perm tube) for validation.

Permeation devices dispense a small amount of gas vapor through a membrane into the sample gas flow stream. The concentration of the analyte is controlled by the permeability of the membrane, temperature, and flow rate of the sample gas. Temperature and flow rate are fixed by the sample system enclosure. Permeability (expressed as ng/min) is a known value stamped on the device. Knowing all these values allows the analyzer to calculate the concentration of the analyte for validation purposes. Typical permeation tube concentrations are 0.5 to 2 ppmv.

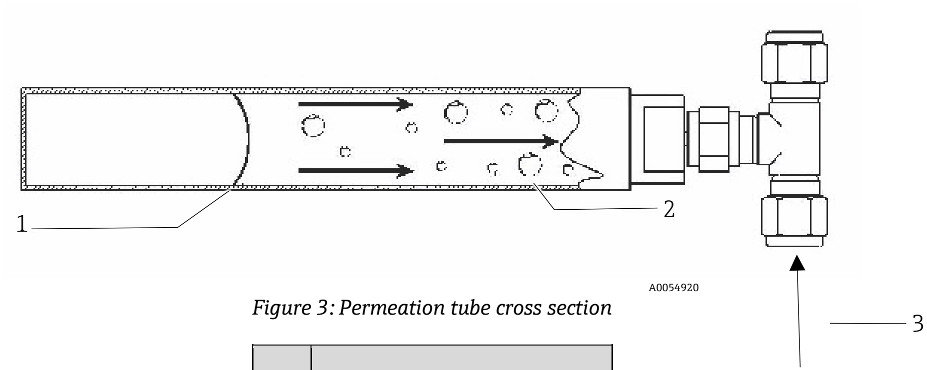


Figure 3: Permeation tube cross section

1	Membrane
2	Water vapor
3	Sample gas flow stream

It can be noted that the sample gas flowing past the permeation device must be free of any pre-existing analyte; therefore, a scrubber or dryer is used to remove the analyte prior to exposure to the permeation device. Due to this, permeation devices are exclusively used on differential measurement TDLAS analyzers as part of an automated validation system.

In general, the validation method can mimic the gas concentration used during production of the analyzer. The validation gas type is listed on each analyzer calibration sheet supplied at the time of order.

4.3 Gas validation design options

When validation gases are used, there are several options to perform the task.

4.3.1 Manual validation

Manual validation relies upon the operator initiating validation through the analyzer electronics and manually opening a 3-way valve which blocks the process gas and allows validation gas to flow to the analyzer.

Products with this option: J22, JT33, SS500, SS2100, SS2100a, and SS2100i.

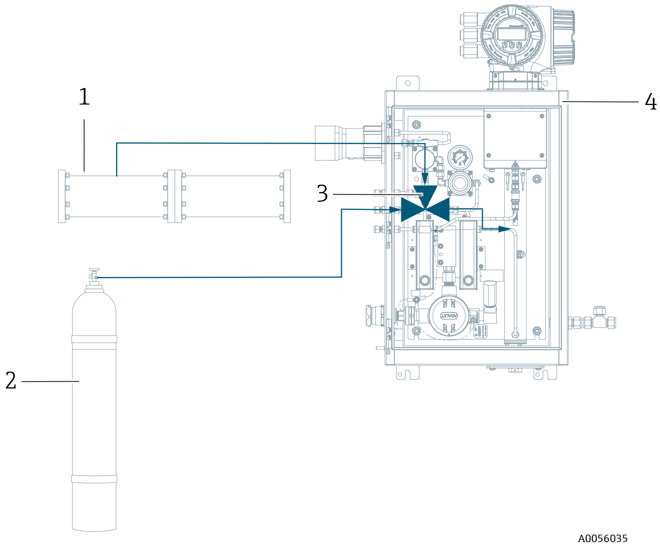


Figure 4: Example of manual validation of a TDLAS gas analyzer

#	Name
1	Process gas
2	Validation gas
3	Manual 3-way valve
4	Gas analyzer

4.3.2 Automatic validation, 1-point

Automatic validation is similar to manual validation however the manual 3-way valve is replaced by either electric or pneumatic air-operated solenoid valves located within the analyzer. Since the actuation of the valve is controlled by the analyzer, the validation can be assigned to start through the human machine interface (HMI) and webserver to perform routine automatic validations at set intervals.

Products with this option: JT33, SS2100, SS2100a, and SS2100i.

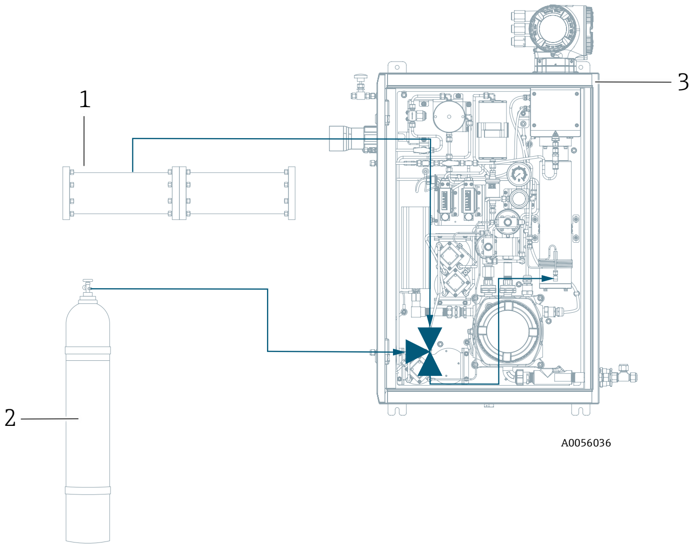


Figure 5: Example of automatic validation, 1-point of a JT33 gas analyzer

#	Name
1	Process gas
2	Validation gas
3	Gas analyzer

4.3.3 Automatic validation, 2-point

This validation design is similar to 1-point automatic validation; however, a secondary validation point can be used for additional verification of the measurement. 2-point validation is often a regulatory requirement for analyzers used in flare monitoring and emissions applications. This option is only offered with pneumatic air-operated solenoid valves.

Products with this option: JT33, SS2100, SS2100a, and SS2100i.

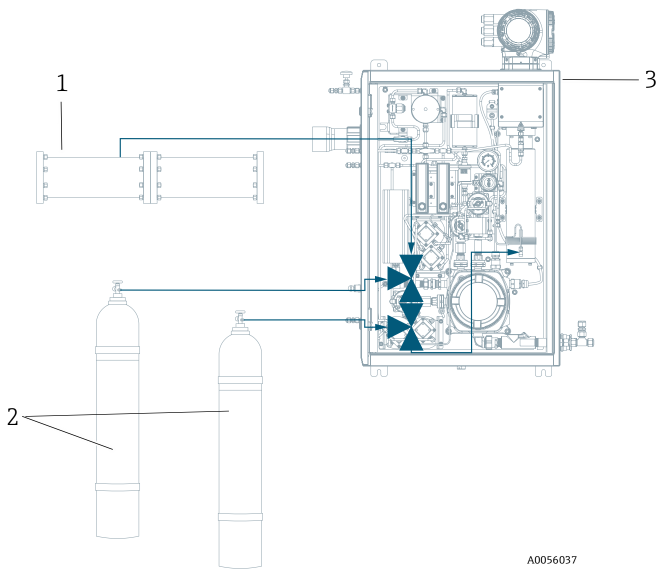


Figure 6: Example of automatic validation, 2-point of a JT33 gas analyzer

#	Name
1	Process gas
2	Validation gases
3	Gas analyzer

4.3.4 Valve options

Automatic validation offers two options for valve actuation: pneumatic and electric. The valve options can be specified within the order code for each analyzer and may be chosen based upon user preference.

Pneumatic solenoid valves are frequently specified for trace measurement applications where tight shut-off is needed. On-site instrument air is required to actuate the valve. Control of the instrument air is handled through separate, additional on-off electric solenoid valves included with the analyzer. Some users prefer the added security and reliability of pneumatic solenoid valves especially if their process demands frequent validations.

Electric solenoid valves are commonly specified in applications where instrument air may not be available. Electric solenoid valves help simplify the sample system design and reduce costs.

4.4 Validation gases

This section focuses on validation using certified gas cylinders. ASTM offers a reliable source for validation of TDLAS gas analyzers. Their standard test methods can be used as reference documents for this purpose. Please refer to the following:

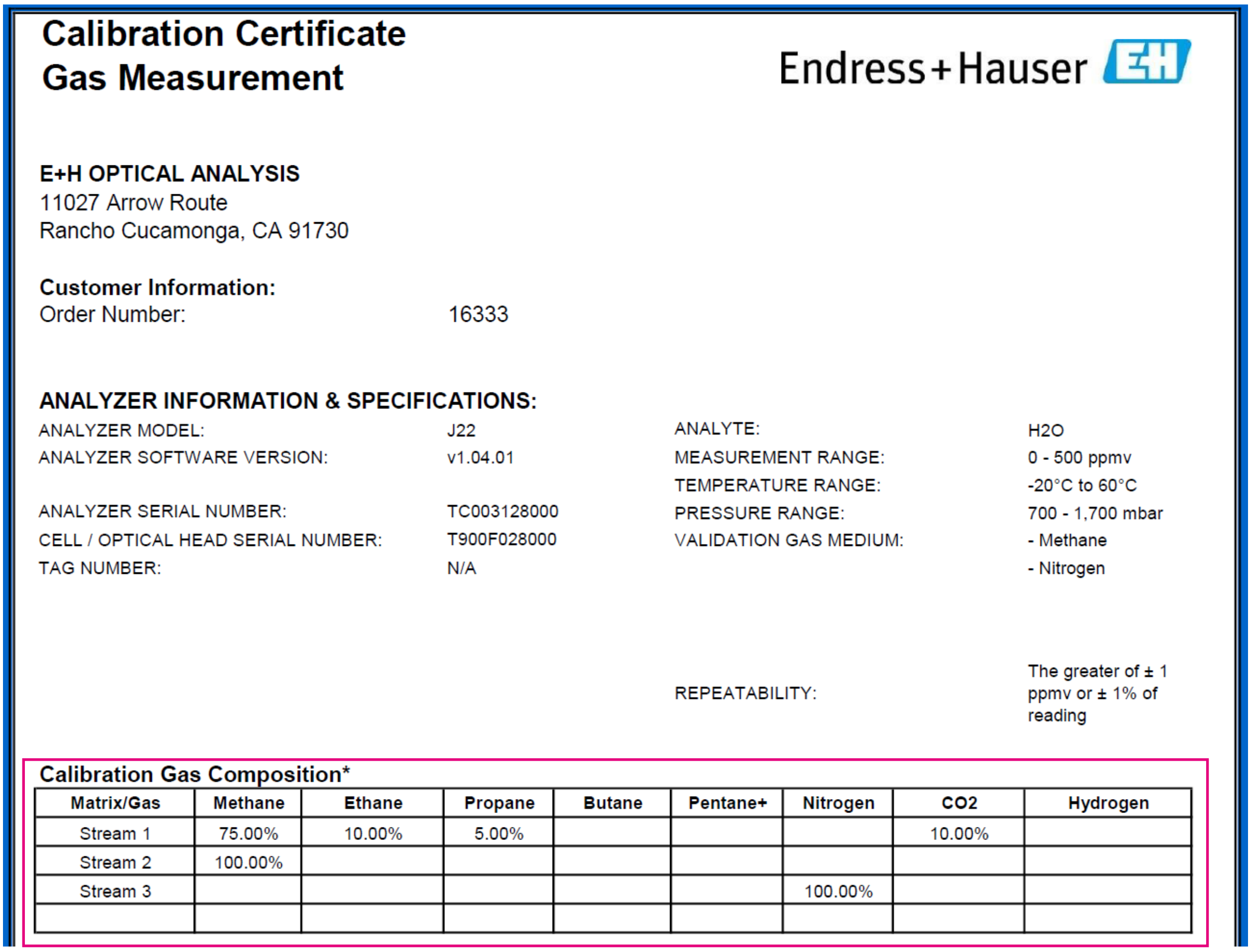
- ASTM D7904 — Standard test method for determination of water vapor in natural gas by tunable diode laser spectroscopy (TDLAS)
- ASTM D8488 — Standard test method for determination of hydrogen sulfide in natural gas by tunable diode laser spectroscopy (TDLAS)

4.4.1 Ranges

The user can refer to the measurement range of their analyzer. Validation gas concentration must fit within this range. Most users choose a validation gas concentration close to their normal operating value. When in doubt, refer to the calibration certificate that came with the analyzer for validation performance or test points.

4.4.2 Backgrounds

The validation gas background can match what is used during factory calibration. Most analyzers have a process stream calibration and a validation gas stream calibration. This information can be found on the calibration certificate provided with the analyzer. For example, a J22 Gas Analyzer uses Stream 1 for process gas measurement in natural gas. Stream 2 would be used for moisture calibration in 100% methane background, while Stream 3 would be used in 100% Nitrogen background. Refer to the figure.



Calibration Certificate
Gas Measurement

Endress+Hauser

E+H OPTICAL ANALYSIS
11027 Arrow Route
Rancho Cucamonga, CA 91730

Customer Information:
Order Number: 16333

ANALYZER INFORMATION & SPECIFICATIONS:

ANALYZER MODEL:	J22	ANALYTE:	H2O
ANALYZER SOFTWARE VERSION:	v1.04.01	MEASUREMENT RANGE:	0 - 500 ppmv
		TEMPERATURE RANGE:	-20°C to 60°C
ANALYZER SERIAL NUMBER:	TC003128000	PRESSURE RANGE:	700 - 1,700 mbar
CELL / OPTICAL HEAD SERIAL NUMBER:	T900F028000	VALIDATION GAS MEDIUM:	- Methane
TAG NUMBER:	N/A		- Nitrogen
		REPEATABILITY:	The greater of ± 1 ppmv or ± 1% of reading

Calibration Gas Composition*

Matrix/Gas	Methane	Ethane	Propane	Butane	Pentane+	Nitrogen	CO2	Hydrogen
Stream 1	75.00%	10.00%	5.00%				10.00%	
Stream 2	100.00%							
Stream 3						100.00%		

Figure 7: Calibration gas composition

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4.4.3 Best practices

⚠ WARNING

Gas cylinders are frequently at high pressure.

▶ Use extreme caution when handling the gas cylinders and field pressure reducing pressure regulators.

There are best practices for sourcing, storage, and utilization of gas cylinders.

- Validation gases can come with NIST traceable certificates. The gases can be of high purity with typical deviation no more than ± 2%.
- Temperature changes can impact gas concentration. Gas cylinders can be stored indoors at room temperature conditions. In general, lower ambient temperatures have a larger effect on validation gases.
- Pressure regulators can be either multi-stage (preferably 4-stage) or heated.
- Tubing from the gas cylinder to the analyzer can be electropolished stainless steel.
- Gas cylinders normally have expiration dates. Do not validate with old gases.
- Gas bottle pressure can be > 13.8 bar (200 psig). A pressure lower than 13.8 bar (200 psig) can result in noisy readings and possible failed validation.

4.5 Validation procedure - example J22 gas analyzer manual validation

This portion of the document reviews a typical manual validation process using gas cylinders. The validation gas can be connected to the validation or reference gas port on the side of the analyzer. The validation gas can be pressure regulated to 15 to 70 kPa (2 to 10 psi).

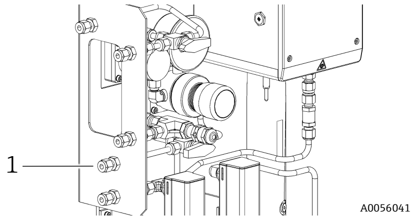

Figure 8: REF GAS IN is the validation gas inlet bulkhead for J22 (1)

4.5.1 Validation configuration

Gas validation settings are found in the Expert menu under Heartbeat technology. Configure the validation settings based upon your application and the validation gases being used. The configuration can be done through the display HMI or through the webserver. The example below shows the webserver configuration.

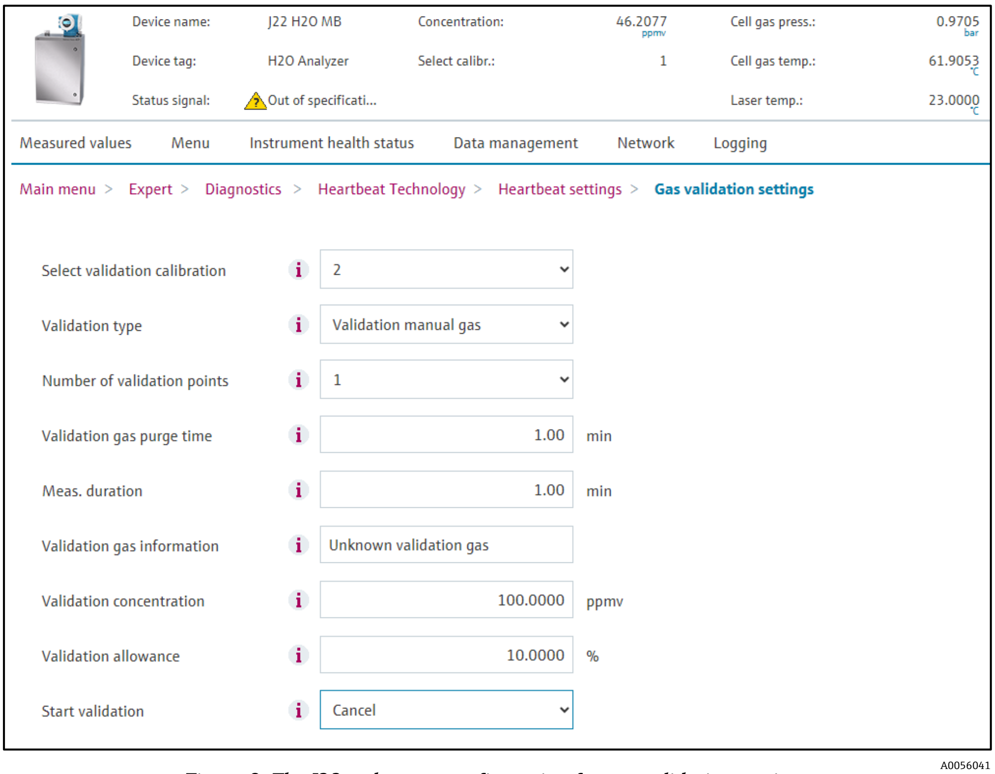

Figure 9: The J22 webserver configuration for gas validation settings

- **Select validation calibration:** This setting identifies the stream that corresponds with the validation gas background. For example, a standard J22 uses Stream 2 for 100% methane backgrounds, Stream 3 for 100% Nitrogen backgrounds. Validations in streams that do not match the validation gas background will result in larger error.
- **Validation type:** Manual or automatic gas validation (analyzer dependent)
- **Number of validation points:** 1 or 2 points (analyzer dependent)
- **Validation gas purge time:** The purge time when switching from process gas to validation gas. Purge time can be set to at least 5 minutes and up to 30 minutes for < 50 ppm applications. In addition, purge time can be increased if the validation gas and valve controlling its flow is far away from the analyzer.
- **Measurement duration:** The amount of time that the validation gas is measured. The measurement duration can be at least 5 minutes (J22) up to 30 minutes for differential TDLAS (JT33 / SS2100).
- **Validation gas information:** This is a user dependent field where the validation gas may be identified.
- **Validation concentration:** The concentration of the validation gas must be identified in this field. This is normally on the certificate from the validation gas bottle or a value from a secondary measurement device, such as a portable gas analyzer or chilled mirror.
- **Validation allowance:** The validation allowance is a percent of the validation gas concentration. During validation, the analyzer will measure gas concentration to determine an average value over the full duration of

the test. If this value falls within the validation allowance, then gas validation will pass. If the measured concentration is outside of the validation allowance, then the gas validation will fail. The recommended default validation allowance is 20%. This accounts for any variations in gas bottles over time, the accuracy of the analyzer, and other external sources of error.

- **Start Validation:** Start or cancel the validation with this selection. Validation gas valve can be opened prior to selecting start validation.

4.5.2 Validation start-up and process

⚠ WARNING

Do not exceed 0.7 barg (10 psig) through the validation port. Damage to the analyzer may result.

1. Check validation gas pressure. Gas can be regulated to 15 to 70 kPa (2 to 10 psi) for J22.
2. Open multi-stage regulator at the gas cylinder to allow gas to flow to the reference gas inlet port on the analyzer.
3. Slowly open the 3-way valve to allow the validation gas to flow through to the measurement cell.
4. Initiate the **start validation** from the web server menu as described above. The J22 analyzer follows the purge and measurement setting that are programmed within the **gas validation settings** page.
5. Check the instrument health status to determine if validation has passed or failed. A passing validation can have the measurement fall within the validation allowance set for the analyzer.
6. If validation has passed, then close the reference gas 3-way valve to allow the analyzer to return to process gas measurement.

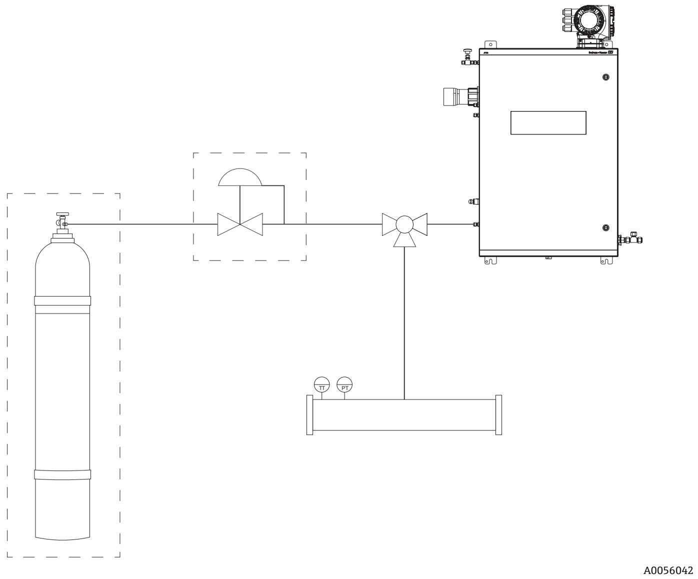

Figure 10: A simple block diagram showing a validation gas standard installation

4.5.3 Validation pass / fail criteria

Passing validation is dependent upon the following:

- Certified validation gas must be used. Gas should have NIST traceable certificate with deviation no more than ± 2 %.
- Multi-stage gas regulator (or heated regulator) is used to reduce gas pressure down to the recommended value for the analyzer.
- Validation is performed on Stream 2 (for methane background) or Stream 3 (for nitrogen background).
- Validation concentration matches concentration of the validation gas bottle or secondary measurement device such as a chilled mirror.
- Validation allowance is set for 20 % or greater.

Please note, passing validation is dependent upon the average gas concentration fitting within the validation allowance set by the user. Gas cylinders can exhibit different concentrations than the published values found on the certificate. Bottle test data from incoming inspection in our factory shows deviations > 10 % are common when verified against chilled mirror moisture analyzers.

The best practices for gas cylinder maintenance listed in this document can be followed to ensure the analyzer passes validation. A failed validation is noted on the analyzer HMI and webserver as shown below. If multiple failed validations occur, a review of the gas validation settings can be conducted. Adjusting the purge time, validation duration, and allowance may help to complete a successful validation.

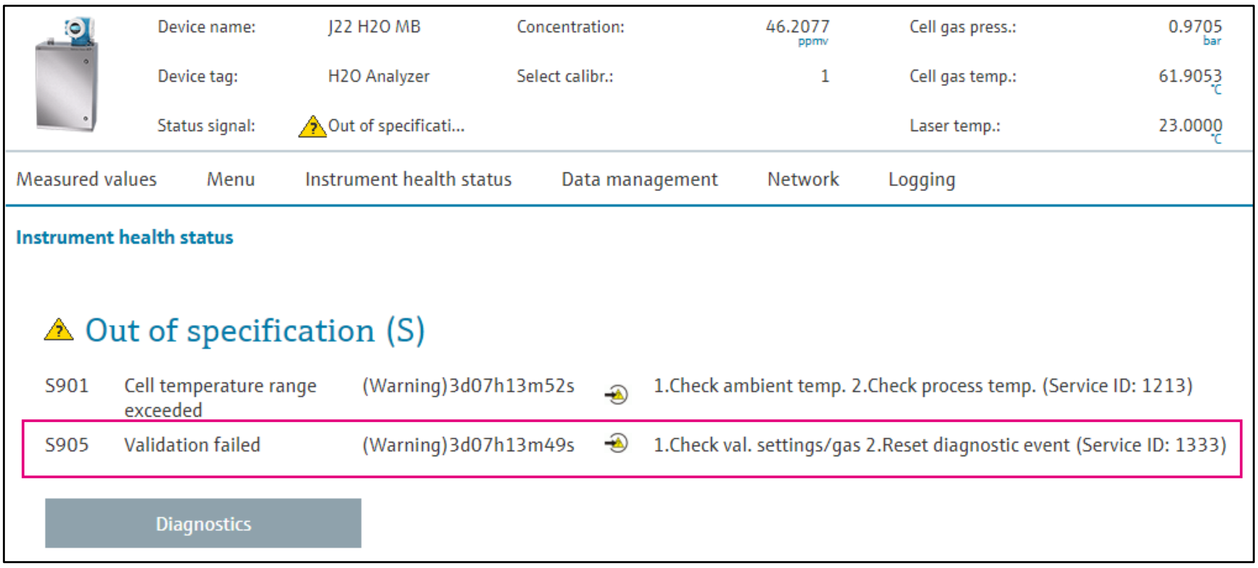

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Figure 11: The J22 webserver health status showing a failed validation

4.5.4 Troubleshooting

If multiple failed validations occur there are several ways to troubleshoot.

- Gas validation settings can be reviewed. Lengthen the purge time, validation duration, and expanding the allowance may help to complete a successful validation.
- Consider the gas cylinder. If the gas cylinder does not meet the best practices described in this document, then it can be replaced by another bottle. Please note any changes to concentration and update the settings within the analyzer.
- A portable gas analyzer may be a helpful tool for troubleshooting. The portable analyzer can be used to check the concentration of the validation gas. If the portable analyzer reads close to the value of the analyzer under test, then the validation gas and related installation can be reviewed.
- If the validation continues to fail, then a zero gas check may be used as a diagnostic test for leakage (applies to moisture analyzers only).

Zero Gas Check for J22 TDLAS moisture analyzers

- A moisture trap can be installed on the validation gas tubing to the analyzer. The trap can be installed in a bypass configuration with isolation valves to prevent continuous exposure to moisture. Endress+Hauser Optical Analysis recommends the Alltech Hydro-Purge II (P/N 14625).
- Isolate the process gas flow using the 3-way valve and flow validation gas through the moisture trap into the analyzer.
- Within thirty minutes, the moisture concentration can fall to < 5 ppm. If higher values are displayed, then they can be recorded and reported to Endress+Hauser Optical Analysis factory service.

4.5.5 Frequency

As TDLAS analyzers offer long-term measurement stability, the frequency of validation is dependent upon the user and their process requirements. Some processes have mandated validation intervals for regulatory compliance. If no interval is required, then a quarterly validation of the analyzer may be used to ensure proper operation.

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