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H_2 Fuel Type I Grade D* Sampling & Analysis ISO-14687:2019 (E) Product Specs

Presented at the International Technical Cleanliness Forum & EXPO "Parts4Assembly"

May 2023

* Gaseous H_2: FCEV Fuel Cells for road vehicles

Mark Taylor, Don Pachuta Ph.D., & Chandler Stryker

Why Test H_2 Fuel?

- Impurities in H_2 Fuel can reduce vehicle efficiency and potentially cause **permanent, expensive** fuel cell damage

- It is the responsibility of the **H_2 Fuel supplier** to identify, measure, and control the levels of impurities listed in the ISO/SAE guidelines to deliver an appropriate ISO/SAE H_2 fuel Grade Product for customer end use

- **TRUST BUT VERIFY.** It is also a responsibility of the **Fuel Station owner and Government** to ensure that the H_2 fuel stored and delivered to customers meets ISO/SAE Purity standards for the intended transport vehicle application.

The Critical Step in <u>ALL</u> H_2 Purity Test Programs is to ensure that a representative H_2 Fuel sample is taken and preserved until analyzed.

3 Key Aspects of an ISO/SAE H_2 Fuel Quality Program

- **Representative Sampling**
- **Safe & Proper Hazmat Shipping**
- **Proper ISO-17025 Laboratory Analysis for Critical Impurities**

Future Goal = Rapid, On-Site Analysis at H_2 Fuel Manufacturers and Fueling Facilities

ISO / SAE Type I, Grade D Purity Specifications & Analytical Methods

Parameter	Guideline Limit	Method
Hydrogen Fuel Index (H_2 Purity):	99.97 % v/v min.	
Total Non-Hydrogen Gases:	300 ppm v/v max.	
Maximum Concentration of Individual Contaminants		
Water (H_2O):	5 ppm v/v max.	ASTM D7653, D7649, D7941, D7941M
Total Non-Methane Hydrocarbons (TNMHC; C_1 Equivalent):	2 ppm v/v max.	ASTM D7675, D7833
Oxygen (O_2):	5 ppm v/v max.	ASTM D7649, D7607
Methane (CH_4):	100 ppm v/v max.	ASTM D7653, D7675, D7833, D5466
Helium (He):	300 ppm v/v max.	ASTM D7833
Nitrogen (N_2):	300 ppm v/v max.	ASTM D7649, D7833
Argon (Ar):	300 ppm v/v max.	ASTM D7833
Carbon Dioxide (CO_2):	2 ppm v/v max.	ASTM D7649, D7653, D7833
Carbon Monoxide[b](CO):	0.2 ppm v/v max.	ASTM D7653
Total Sulfur Compounds[c] (TSC):	0.004 ppm v/v max.	ASTM D7652, D6228
Formaldehyde (HCHO):	0.2 ppm v/v max.	ASTM D7653, D7941, D7892
Formic Acid (HCOOH):	0.2 ppm v/v max.	ASTM D7653
Ammonia (NH3):	0.1 ppm v/v max.	ASTM D7653, D9741/D7941M
Halogenated Compounds (Halogen Ion Equivalent)[d]:	0.05 ppm v/v max.	ASTM D7892, D7676, D5466
Particulate Concentration	1 mg/kg	ASTM D7650, D7651

H_2 Fuel Impurity Profiles are **highly Feed Source Dependent**

Some impurities are introduced via transport / storage and dispensing (**contaminants**)

Some impurities cause ***temporary*** damage to a FCEV Cell

Some Impurities cause ***permanent*** damage to a FCEV Cell

Current Methods of H35 / H70 Station Sampling

Gaseous Impurities – Sampling Protocols

A) **"Gas Serial" type methods** (Sample H35/70 Pumps). Some types **Do not** require a ½ filled FCEV Vehicle for sampling OR a **large** truck mounted >55L tank). Some models just require the **Manual Pump** Mode @ fixed Pressure with station **venting to Atm**.

- **Method 1: ASTM D7606-17:** (ex. NSP-7606 / JSM-7606. Passivated hardware, 1L x **2-valve** passivated ss cylinder purge / fill. **Manual pump mode Only.** Accessories for on-site Detector Tube tests / H_2O / O_2 analyzers + other analytical sensors, JSM-7606 model for sampling of H35/H70 stations

OR

FCEV vehicle tanks, storage tanks, transfer lines, tube trailer applications. Minimal cylinder treatment needed with lab-based prep/testing. All units stored in a ***portable case*** for easy field transport & shipping.

Current Methods of H35 / H70 Station Sampling

Gaseous Impurities – "Gas Serial" Sampling Protocols (Cont'd)

- **Method 2** (Table mounted, non-passivated hardware, 5L x 2-valve Al cylinder). Accessories for On-Site H_2O / O_2 analyzer testing). **Manual pump mode Only**.

- **Method 3:** ISO19880-1 Annex K. Employs **EITHER** a ½ filled FCEV vehicle (& **Auto**-mode)

or **No FCEV vehicle/Tank** & **Manual** mode apps. Uses **1-Valved, large 46.7L** Polished, **1-Valved** Manganese Steel Cylinder (initially evacuated – then filled-flushed-filled several times).

- **Method 4:** Pumps can **EITHER** be in **Auto Mode with a 55L** cylinder to **simulate** a FCEV vehicle or **Manual Mode**. Accessories can allow connected H_2O/O_2 etc. on-site analyzers.

Vehicle manufacturers have recently stated FCEV use during Auto-Mode H_2 sampling is not acceptable - as filling an FCEV with potentially contaminated H_2 may cause permanent cell damage.

Therefore, use of the pump's Auto-Mode for H_2 sampling will mandate use of a large Ballast tank to simulate the FCEV filling process.

Current Methods of H35 / H70 Station Sampling

Gaseous Impurities – Sampling Protocols (Cont'd)

B. "Gas Parallel" **type methods** (**Requires** either a ½ filled FCEV Vehicle **or** a large ballast tank ex. **55+L for sampling**). H35/70 Pumps can be in Auto Mode or Manual Mode. Sample cylinder in parallel line to Vehicle via **T-fitting**.

- **Method 5: ISO19880-1** (**10L x 1-Valve AL cylinder**. Extensive cylinder preparation / multiple purging protocols). **Requires** a ½ filled FCEV vehicle or large ballast tank & Auto-Mode.
- **Method 6: ISO19880-1 + SAE J2601.** Can be used **with / without** ½ filled FCEV Vehicle or large buffer tank. **Auto or Manual** Sample Pump mode. 3 modules. 10L 1-valve Al cylinders or up to 3 x 2.5 – 10L x 2-valved cylinders. Extensive cylinder preparation and protocol.

Vehicle manufacturers have recently stated FCEV use during Auto-Mode H_2 sampling is not acceptable - as filling an FCEV with potentially contaminated H_2 may cause permanent cell damage.

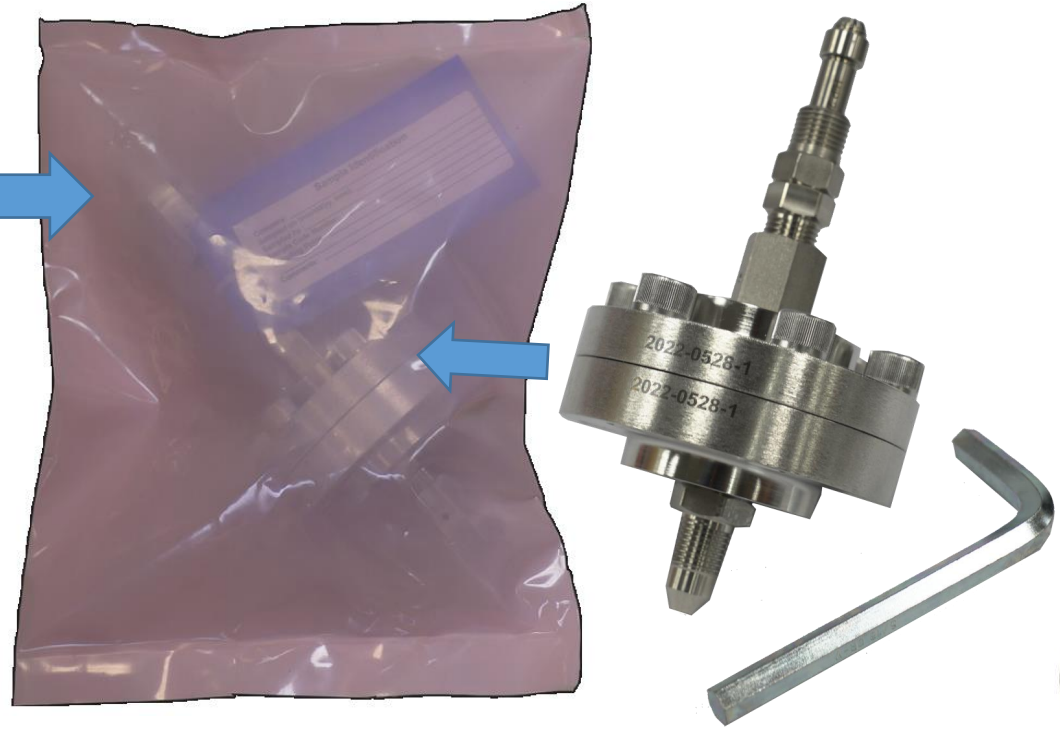
Therefore, use of the pump's Auto-Mode for H_2 sampling will mandate use of a large Ballast tank to simulate the FCEV filling process.

ALI H_2 Fuel Sampling / Shipping Kit (used with NSP/JSM-7606 + MSM-7650) Samplers)

- **2 x 1L Silconert™ Passivated Cylinders**
- **2 x VHP Particulate SS 47 mm Filter Assemblies***

***(45 mm PTFE x 0.2 um PTFE/PE Filter Patches + Frit Backing)**

End-capped & ID Tag Labeled Filter Assy in Anti-static bag



Small, rugged shipping case.

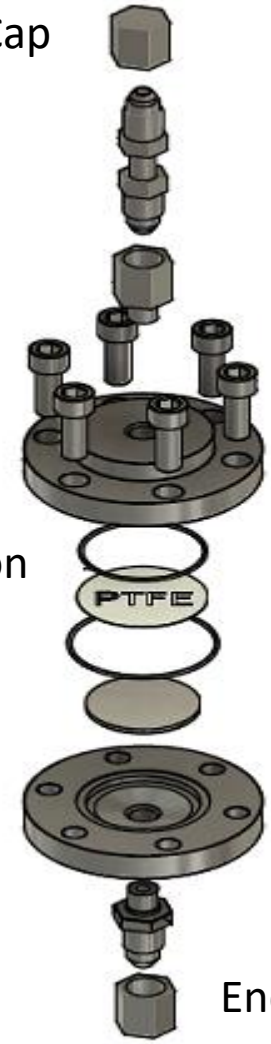
H_2 Fuel Sampling - 1L Cylinder

- All gas wetted parts are Silconert™ passivated to prevent trace impurity wall absorption by active analytes (ex. TSC) + longer life in H_2 service.
- Passivated high quality needle valves with wide seat base.
- Lanyard End-Capped, **Quick Connect** inlet / Outlet valves for NSP / JSM – 7606 Sampler coupling
- 1900 psig (129 bar) Pressure Relief Combination Valve.
- DOT or Pi marked Cylinder bodies.
- Small US Football size for easy transport & air shipping
- Sample Filled to 1,000 psig (68 bar). Many replicate analyses can be performed with this sample size.
- Simple, rapid cylinder prep & on-site purging protocol

2 Cyls provided for Duplicate testing & result verification

H_2 Sampling Kit
Particulate (NVR) VHP SS Filter Housing

(Easily field exchangeable for Multiple Fuel Station Sampling)



H_2 Flow Direction

2-layer Filter Patch PTFE & PE backing

Pre-Weighed, 47mm Filter PTFE x 0.2 um Filter Patches + backing Installed as per ASTM D7650 Method

Current Methods of H35 / H70 Station Sampling

Particulate Impurities; (ex. Non-Volatile Residues (NVR))* by ASTM D7650-21

- **A)** Weighed 0.2 um x 47mm PTFE Filter patch + sampled H_2 wt **calculated** from pressure / temp measurement from a **v. large (55+L) truck-mounted ballast tank .**

- **B)** Weighed Filter patch plus total sampled H_2 kg wt needed for mg/kg H_2 wt measurement is **precisely determined** with by a Coriolis **Mass Flow Meter (MFM)** in **Totalizer** Mode (ex. MRM-7650).

Flow rate typically 10 – 20 g/sec.

Typical FCEV Fuel Tank

*Ex. wear metals, dirt, dust, elastomeric debris, grease, oils

H_2 Sampling Platforms
NSP 7606, JSM-7606* & MRM 7650

NSP-7606 for Gaseous Impurities

MRM-7650 + MFM Totalizer Module for Particulates (NVR)

*Model **JSM-7606** similar to NSP model but designed for sampling **either** H_2 Fuel Stations or H_2 Storage Tanks **or** Sample Lines / Tube Trailers

Proper H35/H70 H_2 Sampling Protocol

Whenever BOTH Gaseous impurities AND Particulates (NVR) must be sampled:

- **ALWAYS perform Particulate sampling 1st** – followed by Gaseous Impurities sampling 2nd

WHY?

- **NVR is typically <u>NOT</u> distributed homogeneously throughout the H_2 gas phase as volatile gaseous impurities & contaminants will be.**

- Typically NVR materials are heterogeneously* surface located (mainly on the interior surface walls) throughout the pump's hardware during a static H_2 gas phase period.

* Because of this physical factor - the **reproducibility** of replicate NVR samples should **not** be expected.

In theory then the 1st NVR sample taken will ***most likely be*** much **higher** than subsequent replicate NVR samples.

NVR sampling is most typical for new stations or after station maintenance actions

H_2 Fuel Sampling for NVR by MRM-7650

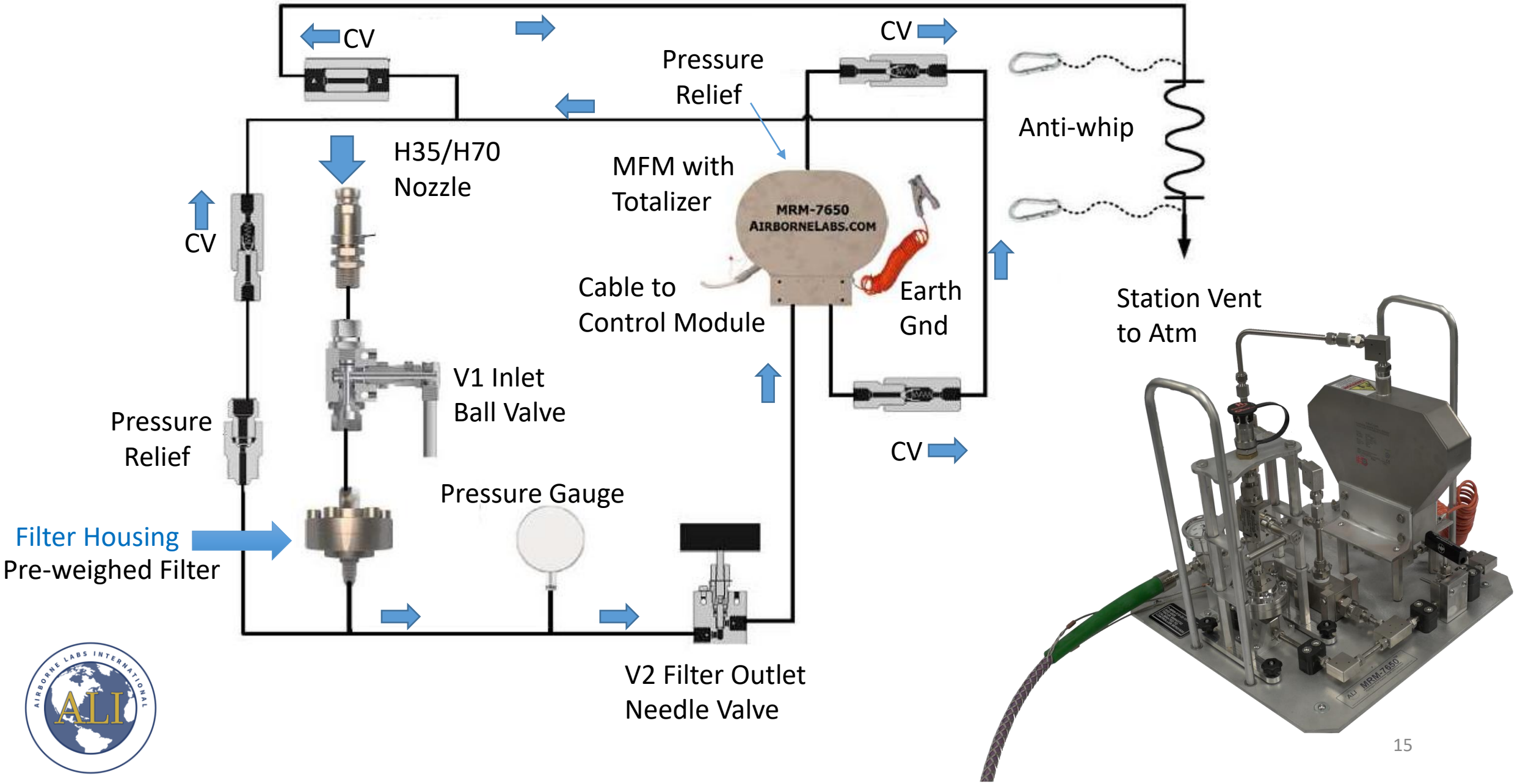
- Designed to sample for NVR **directly** from H35/H70 dispenser nozzles
- Uses a rugged Mass Flow Meter (MFM) with a **Totalizer** for precise and accurate measurement of total H_2 mass & volume sampled.
- Ideal for rapid (<10 min) station sampling

- Fully meets ASTM D-7650-21 requirements for H35/H70 particulate (NVR) sampling.

- Max MFM Flow ≈ 120 g/sec

- Typical Sampling Flow 10 - 20 g/sec

MRM-7650 Gas Flow Diagram - Overview



MRM-7650 H_2 Fuel NVR Sampling

BASIC SAMPLING STEPS – Overview

1) Place Station Dispenser in **Manual** Mode

2) Connect the MRM Ground Wire, MRM Vent line, Pump Nozzle & connect MRM Control Module

3) Pressurize to ≈2,500 psig (170 bar) & Slowly Open V1 plus **leak check** from Nozzle to closed V2. (note: If a leak is found the system must be shut down / depressurized & a new Filter Assy installed).

4) Close V1 – Step Pressure up to ≈5,000 psig (340 bar) then ***Slowly*** Open V1 and leak check **again** (note: If a leak is found the system must be shut down / depressurized & a new Filter Assy installed).

5) Crack Open V2 **slightly** to allow H_2 to enter MFC – then **quickly** Close V2

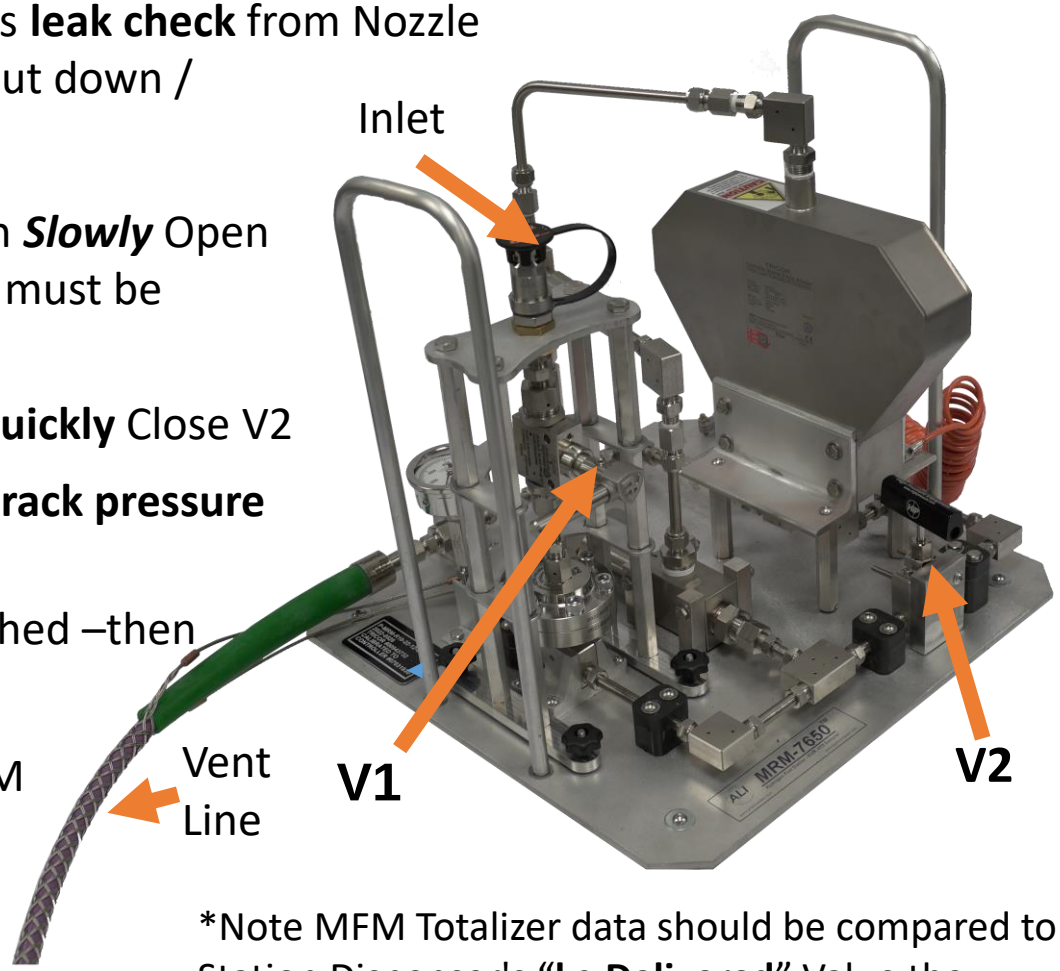
6) Turn on / **Zero** the Control Module **kg Totalizer with CV crack pressure 10 psig (0.7 bar) residual H_2** .

7) Start MCM / Slowly Open V2 until ≈ **15 g/sec** flow is reached –then monitor the **kg of H_2** being sampled.

8) When 2+ kg H_2 **minimum** is sampled – **close** V1 (the MRM will depressurize to minimum CV crack pressure).

9) **Depressurize** the Nozzle Source & Close V2.

10) Remove / Cap the **Filter Assy** + **add kg H_2 Sampled*** To its ID label.

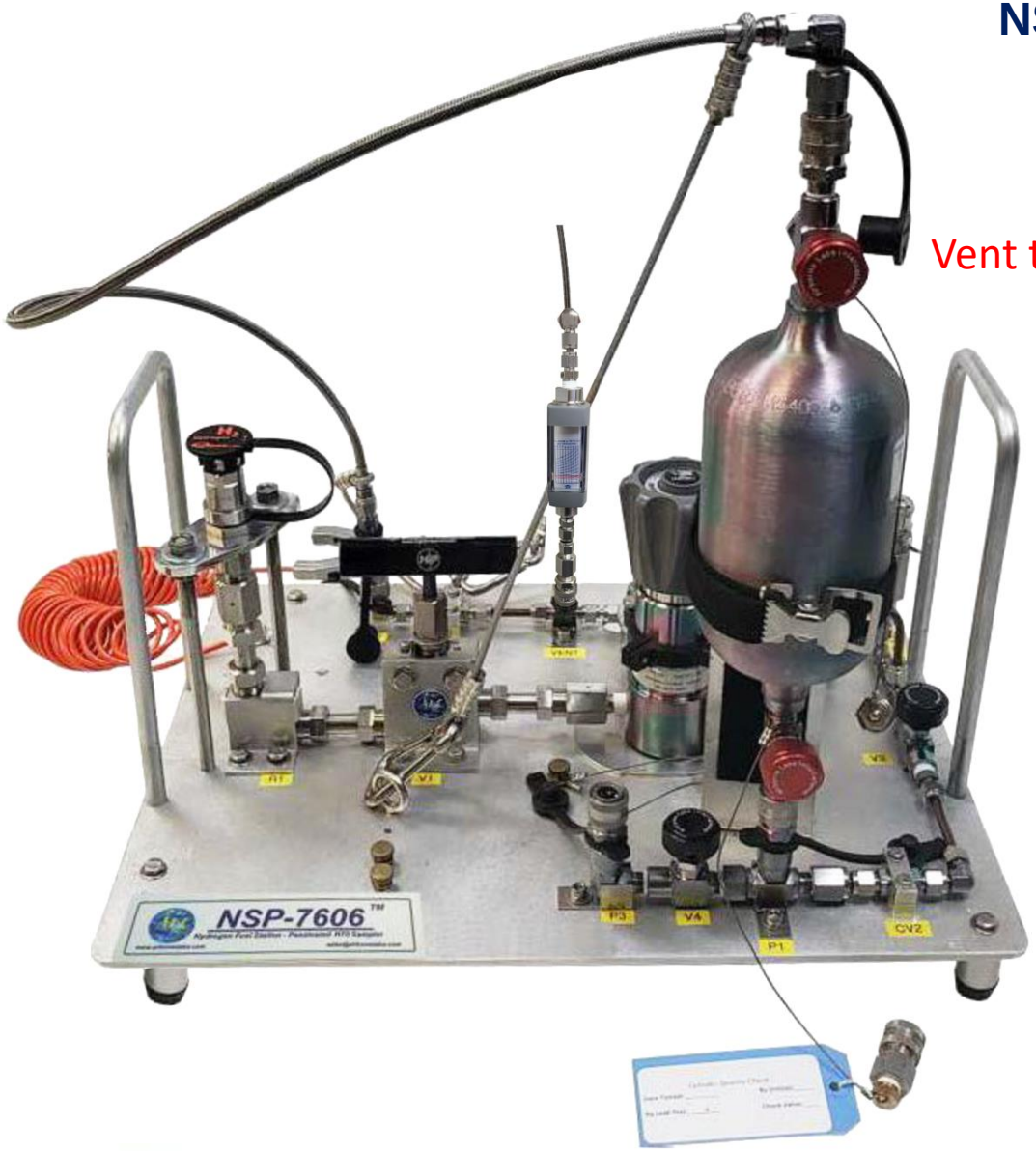


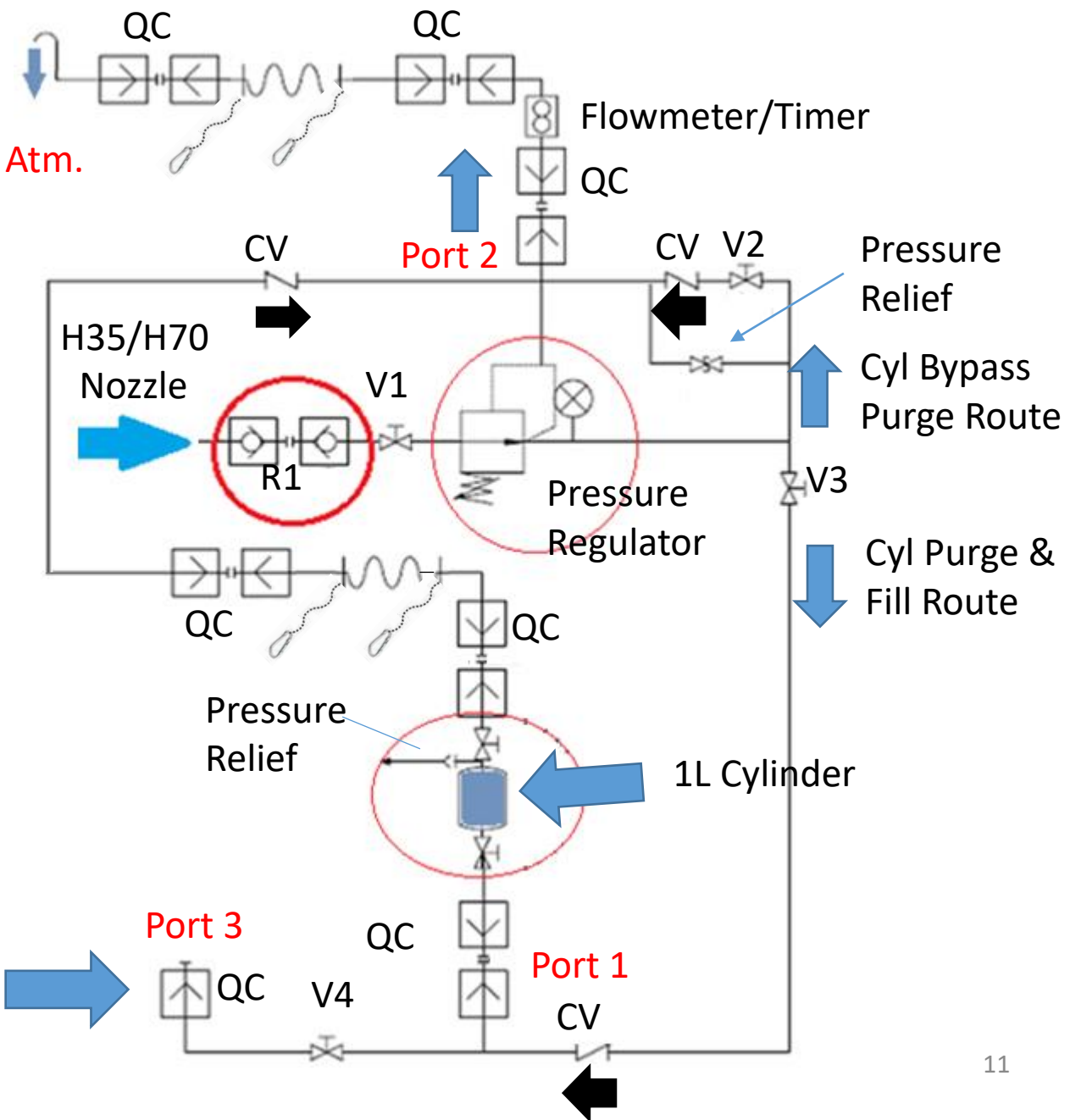
*Note MFM Totalizer data should be compared to Station Dispenser's "**kg Delivered**" Value they **should closely match**.

Gaseous Impurity Sampling

- Gaseous impurities / contaminants should be **homogeneously distributed** in the H_2 Fuel
- Sampling for **Only** Gaseous impurities is **most common** in H35/H70 applications
- Described are 2 Sampler Models: NSP-7606 and JSM-7606 for H35/H70 and many other Sampling applications

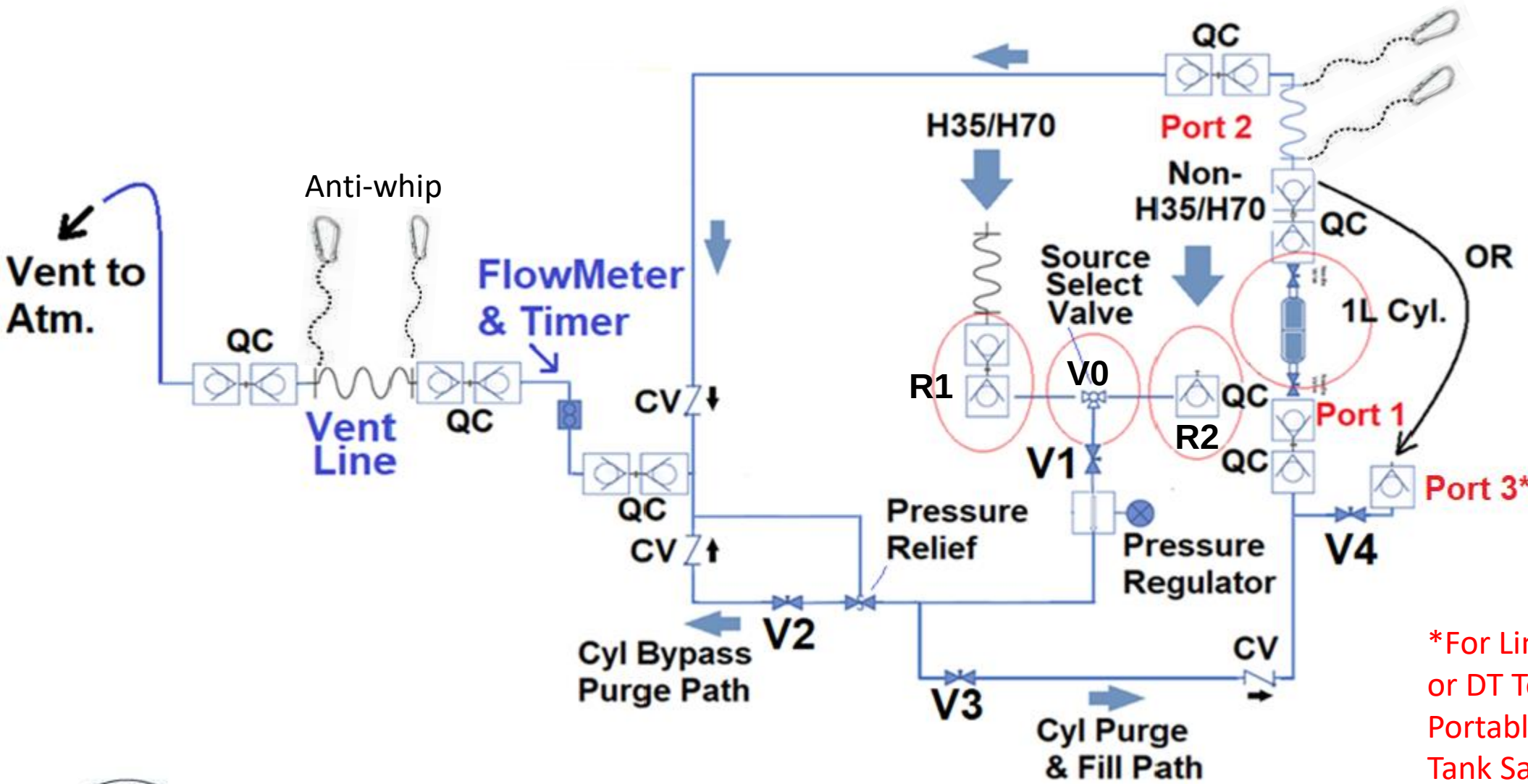
NSP-7606 Gas Flow Plumbing - Overview





For Line Depressurization Or Connect to DT Test Rig /Portable Analyzers / FCEV tank Sampling Accessories

JSM-7606 Gas Flow Plumbing - Overview



Versatile – for Multiple Types of H_2 Fuel Sampling

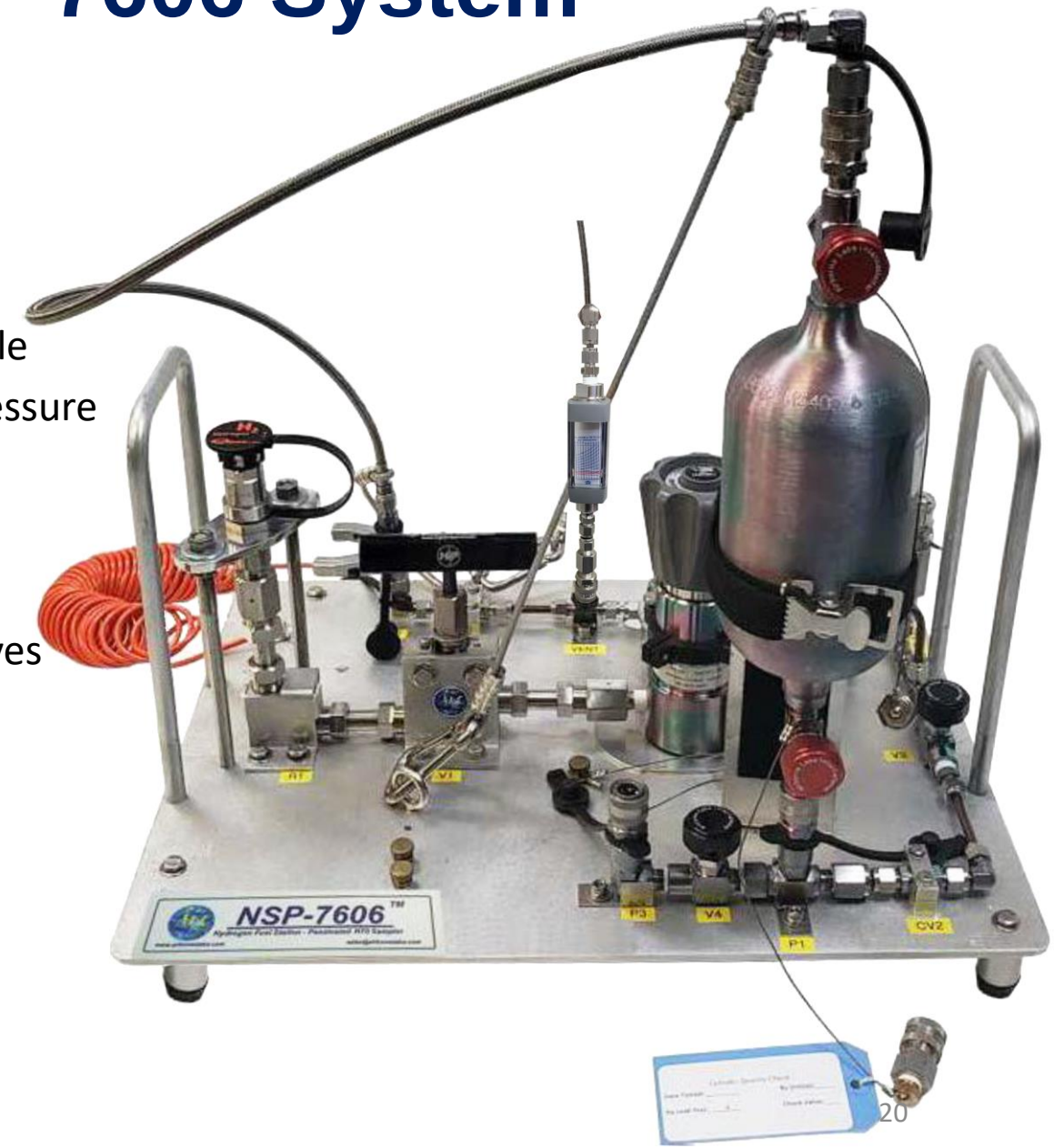
Ex. H35/H70, Pipelines, Storage Tanks, In-Process Mfg., Tube Trucks, FECV's

*For Line Depressurization or DT Test Rig / Portable Analyzers / FCEV Tank Sampling Accessories

H_2 Fuel Sampling by NSP-7606 System

- **BASIC SAMPLING STEPS – Overview**

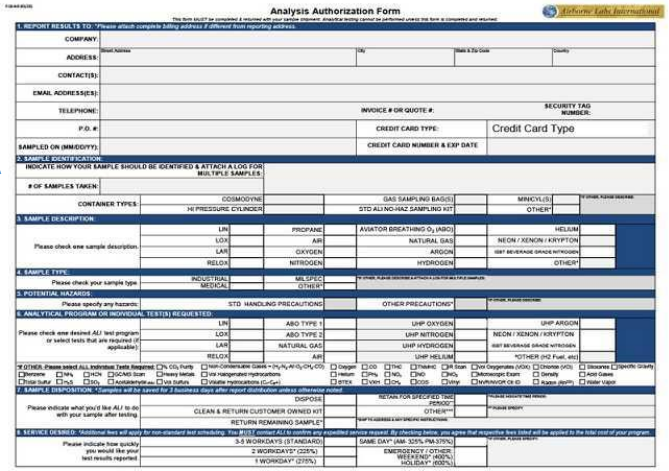
1) Place Station Dispenser in **Manual** Mode
2) **Connect** the NSP Ground Wire, NSP Vent line & Pump Nozzle
3) **Pressurize** Pump to ≈2,500 psig (170 bar) & Set the NSP Pressure Regulator to 1,000 psig (68 bar) & H_2 Leak Test.
4) **Purge** Both Entire Platform Paths
5) **Purge** The 1L Cylinder
6) **Fill** the Cylinder to 1,000 psig (68 Bar) & Close Both Cyl Valves
7) **Depressurize** the pump Nozzle & NSP Platform
8) **Disconnect** / Label / Store the 1st 1L Cylinder
9) **Repeat** Process with the 2nd replicate cylinder



Proper Return of H_2 Fuel Samples (Hazmat Shipment) to ISO-17025 Analytical Lab

Overpack Ship Case

End-capped/ID Tagged Sample Filter Assembly in Anti-static Shipping Bag



Analysis Request Authorization Form

Security Tags

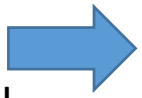
NSP / JSM-7606 + MRM-7650
Portability / Shipping Packages

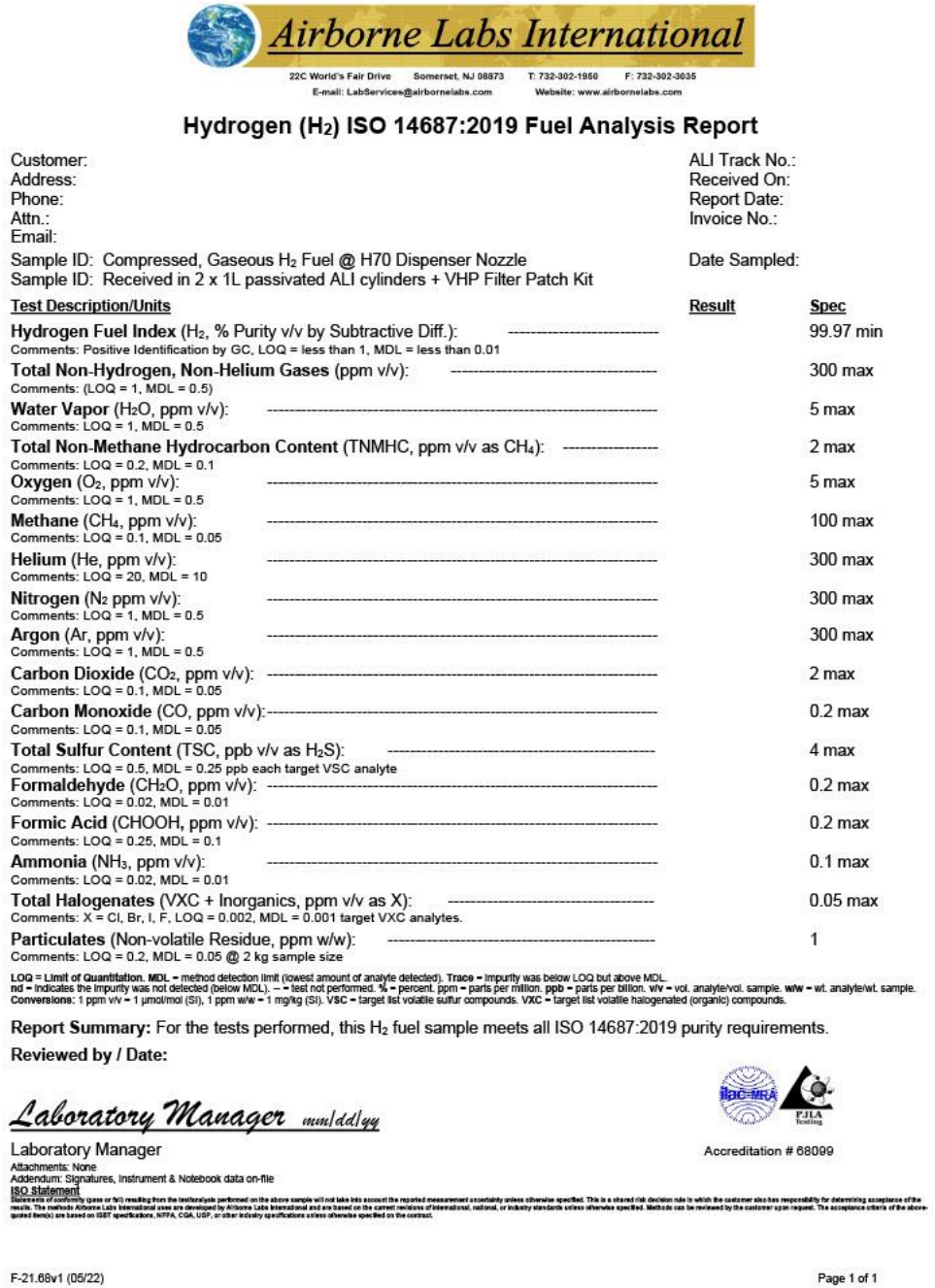
Easily Portable
NSP or JSM or MRM
Samplers in their
Own Rugged
Transport Case

Properly
HazMat-Labeled ALI
H_2 Fuel Sampling Kit

ISO 14687:2019
SAE J2719
COA Report

Reports are Locked and Sealed to prevent potential data tampering





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Hydrogen (H_2) ISO 14687:2019 Fuel Analysis Report

Customer:
Address:
Phone:
Attn.:
Email:

ALI Track No.:
Received On:
Report Date:
Invoice No.:

Sample ID: Compressed, Gaseous H_2 Fuel @ H70 Dispenser Nozzle
Sample ID: Received in 2 x 1L passivated ALI cylinders + VHP Filter Patch Kit

Date Sampled:

Test Description/Units	Result	Spec
Hydrogen Fuel Index (H_2, % Purity v/v by Subtractive Diff.):		99.97 min
Comments: Positive Identification by GC, LOQ = less than 1, MDL = less than 0.01		
Total Non-Hydrogen, Non-Helium Gases (ppm v/v):		300 max
Comments: (LOQ = 1, MDL = 0.5)		
Water Vapor (H_2O, ppm v/v):		5 max
Comments: LOQ = 1, MDL = 0.5		
Total Non-Methane Hydrocarbon Content (TNMHC, ppm v/v as CH_4):		2 max
Comments: LOQ = 0.2, MDL = 0.1		
Oxygen (O_2, ppm v/v):		5 max
Comments: LOQ = 1, MDL = 0.5		
Methane (CH_4, ppm v/v):		100 max
Comments: LOQ = 0.1, MDL = 0.05		
Helium (He, ppm v/v):		300 max
Comments: LOQ = 20, MDL = 10		
Nitrogen (N_2 ppm v/v):		300 max
Comments: LOQ = 1, MDL = 0.5		
Argon (Ar, ppm v/v):		300 max
Comments: LOQ = 1, MDL = 0.5		
Carbon Dioxide (CO_2, ppm v/v):		2 max
Comments: LOQ = 0.1, MDL = 0.05		
Carbon Monoxide (CO, ppm v/v):		0.2 max
Comments: LOQ = 0.1, MDL = 0.05		
Total Sulfur Content (TSC, ppb v/v as H_2S):		4 max
Comments: LOQ = 0.5, MDL = 0.25 ppb each target VSC analyte		
Formaldehyde (CH_2O, ppm v/v):		0.2 max
Comments: LOQ = 0.02, MDL = 0.01		
Formic Acid (CHOOH, ppm v/v):		0.2 max
Comments: LOQ = 0.25, MDL = 0.1		
Ammonia (NH_3, ppm v/v):		0.1 max
Comments: LOQ = 0.02, MDL = 0.01		
Total Halogenates (VXC + Inorganics, ppm v/v as X):		0.05 max
Comments: X = Cl, Br, I, F, LOQ = 0.002, MDL = 0.001 target VXC analytes.		
Particulates (Non-volatile Residue, ppm w/w):		1
Comments: LOQ = 0.2, MDL = 0.05 @ 2 kg sample size		

LOQ = Limit of Quantitation. MDL = method detection limit (lowest amount of analyte detected). **Trace** = impurity was below LOQ but above MDL.
nd = indicates the impurity was not detected (below MDL). -- = test not performed. **%** = percent. **ppm** = parts per million. **ppb** = parts per billion. **v/v** = vol. analyte/vol. sample. **w/w** = wt. analyte/wt. sample.
Conversions: 1 ppm v/v = 1 µmol/mol (SI), 1 ppm w/w = 1 mg/kg (SI). **VSC** = target list volatile sulfur compounds. **VXC** = target list volatile halogenated (organic) compounds.

Report Summary: For the tests performed, this H_2 fuel sample meets all ISO 14687:2019 purity requirements.

Reviewed by / Date:

Laboratory Manager mm/dd/yy

Laboratory Manager

Accreditation # 68099

Attachments: None
Addendum: Signatures, Instrument & Notebook data on-file
ISO Statement
Statements of conformity (pass or fail) resulting from the test/analysis performed on the above sample will not take into account the reported measurement uncertainty unless otherwise specified. This is a shared risk decision rule in which the customer also has responsibility for determining acceptance of the results. The methods Airborne Labs International uses are developed by Airborne Labs International and are based on the current revisions of international, national, or industry standards unless otherwise specified. Methods can be reviewed by the customer upon request. The acceptance criteria of the above-quoted item(s) are based on ISBT specifications, NFPA, CGA, USP, or other industry specifications unless otherwise specified on the contract.

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COA Reports Can be Released

- **Same Day as Received**
- **1 Day / 2 Day / 3 Day**
- **Weekend/Holiday Service**
- **Std = 5 Working Days**

On-Line H_2 Analytical System (HyEx-2719)

(Designed for H_2 Fuel Producers + ISO-17025 accredited H_2 Analytical Laboratory Testing Operations)

- Employs ISO-14687:2019 / SAE-J2719 Recommended Methods and ISO-21087:2019(E) *Validated Equivalent* Analytical Methods (Validation Reports provided with this system)
- Highly automated & **Rapid** Analysis/Test Results With Quality Excursion Alarms
- **Customizable** for Specific H_2 Feed Gas Sources
- Can be used with 1L Cylinder or Transfer Line samples (ex. In-Process or Storage Tanks or Tube Trailers)

H_2 Fuel On-Line Analytical System (HyEx-2719)

Customizable Configuration based on H_2 Feed source

DT Tray

Total Sulfur Pre-Concentrator Module

GC1 for; TSC, CO, CO_2, Ar, O_2, N_2, CH_4

DSA BlackBox Data Station: Sample CH Selection, Impurity List Data Display, System Control, COA Report Generation, QA Alarms, TSC, TNMHC, %H_2 Purity Calcs, Data log.

GC2 for; He, H_2O, THC & others

GAS-2XLH = 2 Ch DT Analyzer: NH_3, Halogens, Aldehydes, Organo / Halo-Acids etc.

AccuCal = Precision Dilution of Stock Stds to Working Cal Stds

ASCM-5H Gas Controller: Sample Ch or Gas Std Selection

H_2 Carrier Gas & Zero Air Generator

Power Conditioner / UPS

Summary

- Proper H_2 Fuel Sampling is a **critical 1st step** in H_2 Purity Testing for proper Vehicle use.
- In all applications, a **Representative Sample** of the H_2 Fuel Must be taken. This is challenging for H_2 Fuel.
- The **Integrity** of the sample needs to be **<u>preserved</u>** from Time of Sampling to Final Analysis**. <u>Short</u>** time periods desired.
- **Several types** of Sampling Systems are available (ex. Gas Serial & Parallel) Each with advantages / disadvantages
- **Sampling Applications:** H35/H70 Refueling Stations, Vehicle Tanks, Bulk Storage Tanks, Pipelines, Plant Transfer Lines, In-Process Plant points, Tube Truck Loads.
- **Proper Hazmat Sample shipping** (when needed) is required.
- **Analysis** by an ISO accredited Lab using validated methods and fast result turn-around is required
- Development of ***Economical, Effective,*** **On-Site H_2 Fuel Analyzers** for use at H_2 Fuel Manufacturers and point-of-use refueling sites is the ***next step*** in assuring Spec. Grade H_2 Fuel.

Thank You – Questions?

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