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Qualification and Pre-Screening of Lubricants for Use in High-Pressure Hydrogen Tanks: Ensuring ISO 14687 Grade D Purity Within Fuel Cell Drive Trains

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Abstract

Fuel cell electric vehicles (FCEVs) require specific hydrogen purity, as even trace contaminants can degrade proton exchange membrane fuel cells (PEMFCs). While hydrogen quality is monitored along the supply chain according to international standards, potential contamination from in-vehicle materials, such as lubricants and greases, remains largely unexplored. Here, we present a staged testing framework consisting of (i) a rapid pre-screening for formulation stability and (ii) a full qualification pathway to assess lubricant-derived contamination under realistic refueling conditions. Candidate lubricants were exposed to hydrogen in a 700 bar Type IV vessel following an SAE J2601 refueling procedure. Contamination risks were evaluated by optical inspection, particulate matter, and gas analysis, monitoring contaminants specified in ISO 14687:2025 Grade D. The applicability of the framework was demonstrated in practical scenarios. In the pre-screening pathway, a silicone-based formulation fulfilled the 24 h acceptance criteria for formulation stability and was classified as potentially suitable for high-pressure hydrogen tank applications. In contrast, two other lubricants based on silicone and mineral oil exhibited visible changes associated with increased risk of particulate matter release, resulting in a classification of unsuitable. In the full qualification pathway, the fluorinated DuPont™ MOLYKOTE® HP-300 Grease was evaluated over 23 days and showed no release of harmful contaminants into the hydrogen gas, leading to the classification of suitable. Collectively, the presented protocols provide a structured basis for screening and qualifying lubricants for high-pressure hydrogen tanks in PEMFC applications, supporting future standardization in hydrogen technologies.

Keywords: fuel cell electric vehicles; FCEV; ISO 14687; lubricants; qualification protocol; hydrogen gas analysis

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1. Introduction

Hydrogen quality is a critical parameter influencing the performance and adoption of emerging hydrogen technologies. Purity requirements are defined in international standards, such as ISO 14687:2025 [1], ISO 21087:2019 [2], ISO 19880-8:2024 [3], and SAE J2719_202003 [4], which specify allowable concentration limits for individual contaminants across various use cases. Embedded in technical guidelines and regulatory frameworks, this

standard plays a pivotal role in ensuring reliability and compatibility across the growing hydrogen infrastructure [5–9].

A prime example of its importance is found in proton exchange membrane fuel cells (PEMFCs), a cornerstone technology for decarbonizing both transportation and stationary power sectors due to their high efficiency, rapid start-up, and compatibility with renewable hydrogen [10–13]. The long-term performance and durability of PEMFCs are highly sensitive to hydrogen purity, with even trace levels of certain contaminants capable of reducing stack lifetime and efficiency. The effects of hydrogen contaminants on PEMFC components are summarized in international norms, such as EN 17124:2025 [14] and several review papers [15–17]. For instance, carbon monoxide (CO), ammonia (NH_3), and hydrogen sulfide (H_2S) have been reported to cause irreversible catalyst poisoning, membrane degradation, or blockage of active sites at the electrodes [15,17–20]. To safeguard PEMFC operation against such degradation mechanisms, ISO 14687:2025 [1] Grade D prescribes strict hydrogen quality requirements, which are enforced through routine monitoring within the hydrogen supply chain, encompassing production, purification, compression, and distribution stages up to the point of dispensing [21–27].

However, despite upstream control, onboard contamination of fuel cell electric vehicles (FCEVs) remains largely unaddressed in current quality assurance protocols. Previous studies have shown that materials used in fuel cell systems, including lubricants, adhesives, sealants, hoses, and structural materials, can release contaminants that affect PEMFC performance and durability [28]. In particular, lubricants, including grease-type formulations—hereafter referred to collectively as lubricants to describe materials commonly applied to seal valves, fittings, and on-tank valves (OTVs)—have been identified by the U.S. National Laboratory of the Rockies (NRL) as potential contamination sources through in situ FC testing [28]. Additionally, lubricating oils, specifically in the presence of water traces, have been reported to facilitate the interaction of hydrogen and metallic tank components, causing increased hydrogen permeation and potential material degradation under thermal stress [29–35]. These findings indicate that lubricants not only threaten the structural integrity of hydrogen storage systems but also affect fuel cell integrity and pose a risk to maintaining ISO 14687:2025 Grade D compliance in the fuel cell drive train.

Against this background, lubricant-induced contamination during refueling represents a specific but rarely addressed pathway by which hydrogen may fail to meet PEMFC purity requirements. In FCEVs, lubricants are typically applied to OTVs and directly exposed to hydrogen at pressures up to 700 bar and temperatures ranging from −40 °C to +85 °C [36]. Under these conditions, lubricants may degrade and contaminate the tank, releasing volatile organic compounds or particulate matter into the hydrogen gas. Such impurities can subsequently enter the fuel cell system undetected, potentially causing damage to the PEMFC stack over time (Figure 1a). While ISO 14687:2025 acknowledges this hazard by requiring refueling stations to prevent contamination from operating fluids [1], standardized and methodologically simple methods for evaluating lubricant suitability for high-pressure hydrogen tanks in FCEVs are currently not available.

In this work, we introduce a staged framework for the assessment of lubricants intended for use in FCEV hydrogen tanks, particularly during refueling. The framework consists of a simplified pre-screening protocol for rapid compatibility assessment and a full qualification pathway for application-relevant testing under realistic hydrogen exposure conditions. Both protocols are based on defined lubricant suitability criteria, including visual inspection and compliance with ISO 14687:2025 Grade D hydrogen purity requirements, and are designed to evaluate hydrogen contamination originating from onboard lubricant sources in Type IV hydrogen tanks (Figure 1b). In both stages, candidate lubricants are applied to stainless steel sinter plates and exposed to hydrogen under SAE J2601

automotive refueling conditions [36]. The pre-screening protocol enables rapid evaluation and early elimination of potentially unstable lubricant formulations within 24 h, whereas in the full qualification pathway, lubricant suitability is assessed over a 23-day dwell period in the hydrogen tank. The applicability of the framework is demonstrated in practical test scenarios, in which finished lubricant formulations with only partially disclosed compositional information were evaluated against the pre-defined acceptance and protocol completion criteria, resulting in classification of the tested lubricants as suitable, potentially suitable, or unsuitable for use in FCEV hydrogen tanks.

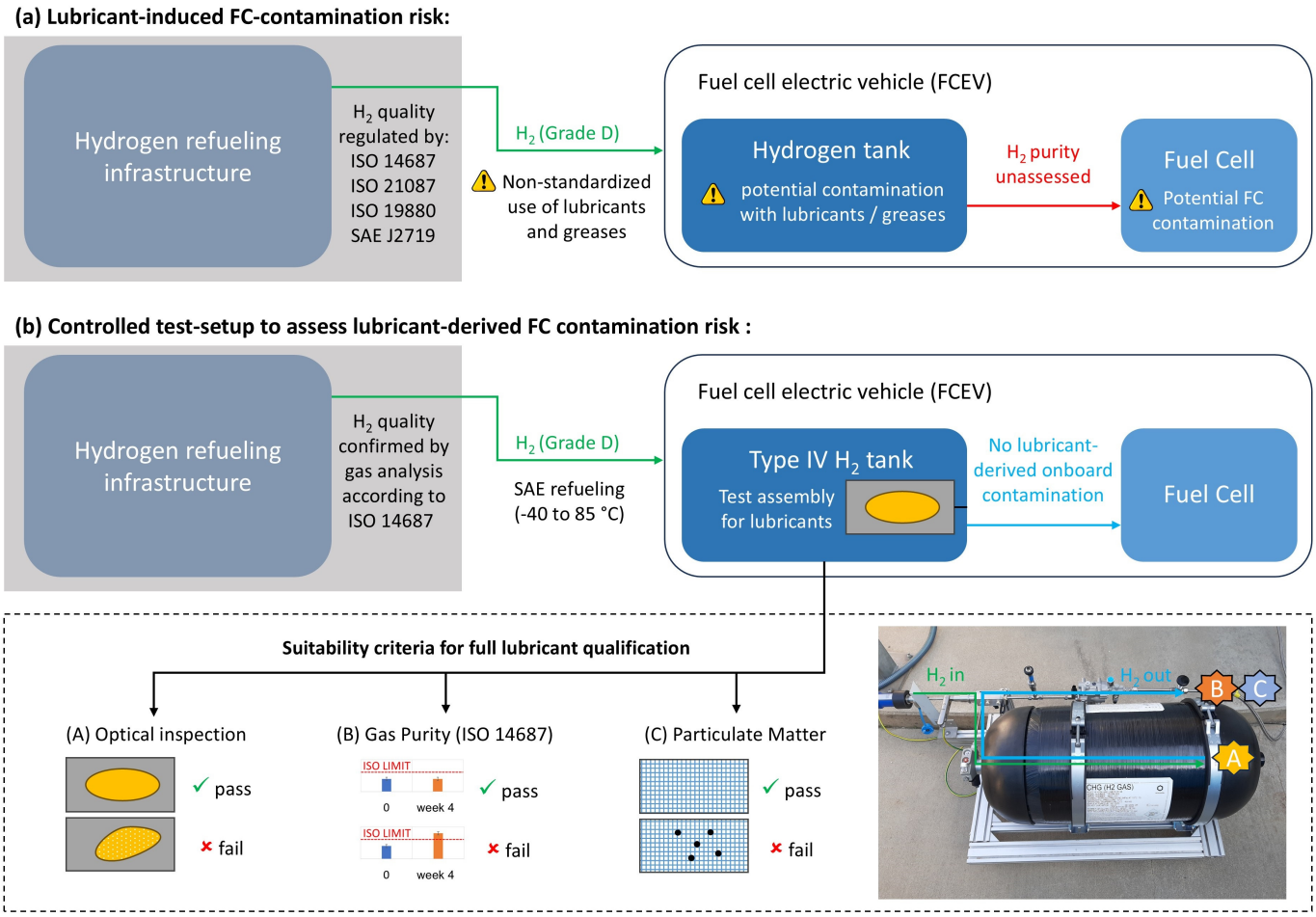


Figure 1. (**a**) Fuel cell contamination risk posed by onboard lubricants; (**b**) schematic of test setup and full qualification protocol to evaluate lubricant suitability in H_2 high-pressure tanks for fuel cell drives [1–4].

Combined, this testing framework provides a structured methodology for ensuring compliance with ISO 14687:2025 Grade D hydrogen quality requirements and supporting the protection of PEM fuel cell performance and durability.

2. Experimental Test Setup

To evaluate the risk of lubricant-induced hydrogen contamination under realistic refueling conditions, a dedicated testing protocol was developed. The test setup employed a commercially available 70 L Type IV hydrogen tank (76 L, Hexagon Purus ASA, Oslo, Norway, maximum allowable filling pressure of 875 bar) equipped with hydrogen-compatible instrumentation for pressure regulation, gas sampling, and in-line purity monitoring. Type IV tanks are characterized by a thermoplastic PA/PE liner and a full carbon- or glass-fiber composite overwrap, providing weight advantages compared with metallic tank concepts

that make them the preferred onboard storage technology for mobile PEMFC applications, including passenger cars, buses, and trucks operating at 350 or 700 bar [37]. Since PEM fuel cells are the most common fuel cell type for vehicle applications, the selected Type IV tank represents the relevant storage technology for the investigated PEMFC-based FCEV use case [38].

Analytic instruments included a Shimadzu AP125WD precision scale (Shimadzu Handelsgesellschaft mbH, Korneuburg, Austria), a Matrix MG 5 real-time Fourier transform infrared (FTIR) gas analyzer (Bruker Austria Gmbh, Vienna, Austria), a combined ion–molecule reaction (IMR) and electron impact (EI) mass spectrometer (ComboSense®, V&F Analyse- und Messtechnik GmbH, Absam, Austria), and a Zeiss Stemi 305 optical microscope (Carl Zeiss GmbH, Vienna, Austria). Detailed descriptions and analytic procedures are provided in Section 3.

2.1. Preparation of Test Assemblies

Candidate lubricants were introduced into the Type IV tank (76 L, Hexagon Purus ASA, Oslo, Norway) using stainless steel sinter plates (AISI 316L (1.4404), ca. 30 × 100 × 5 mm^3), selected for their high mechanical stability and proven material compatibility in hydrogen test systems [39]. Prior to use, plates were precision-cleaned by rinsing with distilled water and cleaning in an ultrasonic bath, where they were fully immersed in isopropanol for 30 min at 80 °C. Afterwards, the sinter plate was dried at 100 °C for 30 min. Cleanliness was confirmed by comparing the pristine and post-cleaning mass of the sinter plate using a Shimadzu AP125WD precision scale (Shimadzu Handelsgesellschaft mbH, Korneuburg, Austria, average of three measurements). For lubricant application, the formulation was first homogenized until no phase separation was observed. Then, 1.000 g ± 0.002 g of lubricant was applied as a thin layer on both sides of the sinter plate using a metal spatula, using special care to avoid the inclusion of air bubbles and film inhomogeneities (Figure 2a). This step was carried out under cleanroom conditions (ISO Class 7 [40]) to minimize the risk of air entrapment, localized concentration gradients, and subsequent non-uniform lubricant degradation during H_2 exposure. The quantity of the tested lubricants was deliberately set to 1 g to simulate a worst-case overlubrication scenario. Since connector and fastener sizes are not standardized for hydrogen refueling infrastructure and FCEV tanks, commercial solutions using ISO M45 [41] threads (76 L, Hexagon Purus ASA, Oslo, Norway, type IV hydrogen tank) were used to guide realistic lubrication scenarios. Based on this reference, an active flank area of approximately 60 cm^2 was estimated. Assuming lubricant densities of 1.0 g cm^{-3} and film thicknesses of 10–50 µm, realistic lubrication requires between 0.06 g and 0.30 g of lubricant. Consequently, the selected 1 g far exceeds typical application amounts, providing a robust safety margin for evaluating lubricant compatibility under conservative testing conditions.

2.2. Integration into the Test System

The sinter plate with the lubricant layer was mounted securely at the rear end of the commercially available Type IV hydrogen storage tank (76 L, Hexagon Purus ASA, Oslo, Norway, Figure 2b). For this, the test assembly (sinter plate with lubricant layer) was suspended on a stainless steel wire attached to a 10 cm stainless steel rod, which was fastened to the threaded port of the tank's end plug. The test assembly was carefully inserted into the tank, preventing physical contact with any external and internal tank components. The position at the rear end was chosen to guarantee direct exposure to pressurized hydrogen during refueling while ensuring reproducible positioning across all test runs. Previous studies on fast filling of Type IV hydrogen tanks indicate that temperature distributions can be non-uniform and that regions near the tank end may

experience elevated thermal loads and pronounced temperature gradients [42]. Thus, this location is selected not only as a representative position in terms of maximum thermal stress, but also because the end plug ('boss') is typically lubricated under standard operating conditions. In this way, the position of the subsequent end-use application is reproduced as closely as possible to its real configuration.

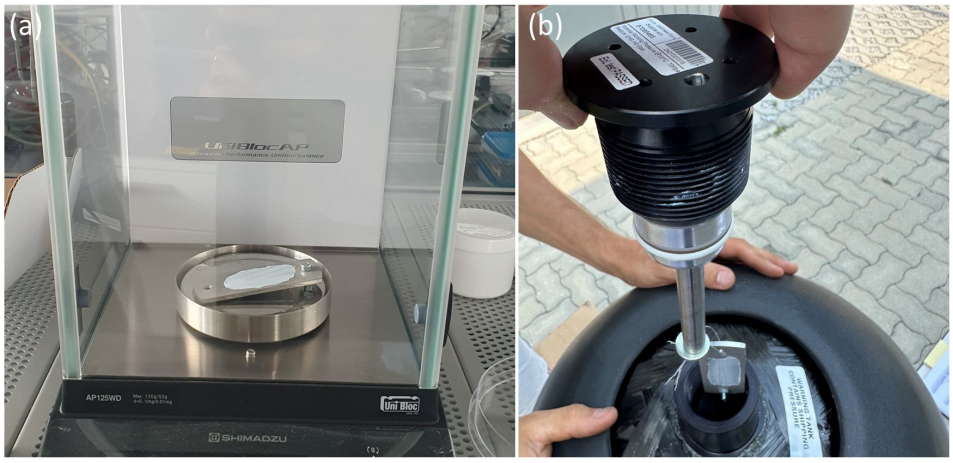


Figure 2. Pictures of the test assembly during (**a**) precision weighing after lubricant application, (**b**) insertion into the test system.

2.3. Hydrogen Refueling

Following test assembly integration, the tank was connected to the refueling infrastructure at HyCentA using a standard OTV setup (H_2 OTV, OMB Saleri S.p.A. SOCIETÀ BENEFIT, Brescia, Italy) according to manufacturer specifications. The system was subsequently purged with nitrogen (Linde Gas GmbH Österreich, Stadl-Paura, Austria, min. 99.998% purity, 2 × 12 bar cycles) to displace residual atmospheric gases and ensure no leakages. This was followed by flushing with min. ISO 14687:2025 Grade D hydrogen and filling the tank to 20 bar (Figure 3a). Afterwards, the test protocol adhered to standardized SAE J2601 refueling profiles [36]. The boundary conditions for the refueling setup are defined by a non-communication (no-com) fueling protocol with an initial pressure of 10 bar, a target final pressure of 742 bar, and hydrogen pre-cooling at –20 °C. These conditions necessitate the application of SAE J2601, Table B-70 [36]. At an ambient temperature of 35 °C, this corresponds to a required pressure ramp rate of 44 bar/min, which was rigorously maintained at the dispenser, where internal tank pressure and temperature were continuously monitored (Figure 3b).

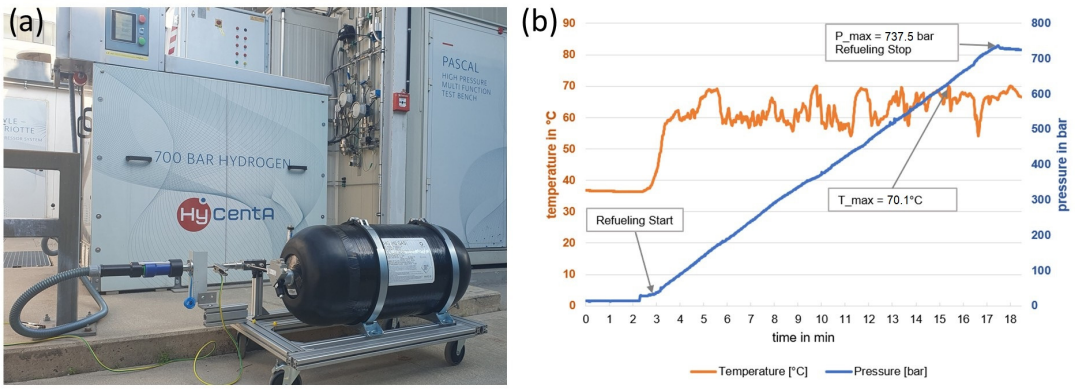


Figure 3. (**a**) Picture of the test system connected at the HyCentA 700 bar refueling infrastructure (HyCentA Research GmbH, Graz, Austria); (**b**) temperature and pressure detected in the tank during refueling aligned with SAE J2601 guidelines.

3. Analytic Measurements

When the target pressure of 742 bar was reached, gas purity was assessed within a 15 to 30 min time window after filling to (i) verify that hydrogen purity (Grade D) was maintained during the refueling process, and (ii) establish a baseline for H_2 quality (t = 0) when evaluating lubricant suitability over defined test periods. This approach ensured that any increase of ISO 14687:2025-regulated impurities detected during the study could be attributed to the presence of the lubricant rather than conditioning or pre-test contamination. Over the test period, the system was held under static conditions. For pre-screening, a dwell period of 24 h was used to investigate lubricant formulation stability. In contrast, for the full qualification protocol, dwell periods of up to 23 days were defined to simulate typical dwell intervals between refueling events (based on 10,000 km/year and approximately 600 km driving distance per refill). Potential leaching, outgassing, or degradation of the lubricant was assessed via the following combined analysis approach.

3.1. Optical and Gravimetric Assessment of Lubricant Films (Test A)

After the protocol dwell period (24 h for pre-screening and up to 23 days for full qualification), the tank was depressurized according to the tank manufacturer specifications, and the test assemblies were retrieved from the tank internals. The uniformity of the lubricant layer was assessed via optical comparison before and after exposure, and lubricant losses were quantified by precision weighing using a Shimadzu AP125WD precision scale (Shimadzu Handelsgesellschaft mbH, Korneuburg, Austria, average of three measurements).

3.2. Gas Analysis Aligned with ISO 14687:2025 Grade D (Test B)

Hydrogen purity assessment was performed in alignment with ISO 14687:2025 Grade D using a Matrix MG 5 real-time Fourier transform infrared (FTIR) gas analyzer (Bruker, Austria Gmbh, Vienna, Austria) and a ComboSense® (V&F Analyse- und Messtechnik GmbH, Absam, Austria), which combines an ion–molecule reaction mass spectrometer (IMR-MS) with an electron impact mass spectrometer (EI-MS).

The FTIR spectrometer contains a mercury–cadmium–telluride detector cooled with liquid nitrogen, and a Rocksolid™ interferometer (Bruker Optics GmbH & Co. KG, Ettlingen, Germany) featuring permanent alignment. A 5 m multi-reflection gas cell is used with an aluminum body coated in nickel, zinc selenide (ZnSe) windows, and gold-coated mirrors. The gas cell is maintained at a temperature of 25 °C. The spectral resolution is below 0.5 cm^{-1}, with a spectral rate of 0.5 cm^{-1}/s. The FTIR's housing, containing the beam path outside the gas cell, is enclosed in a Zarges® box (ZARGES GmbH, Weilheim, Germany) and is continuously purged with nitrogen at a flow rate of 800 mL/min. This purging creates an environment in the beam path that is free from environmental gases. The FTIR system is designed to provide high-precision quantification of gaseous compounds, even at very low concentrations, without requiring calibration. Detection of analytes is achieved using reference spectra provided by the manufacturer (Bruker Austria Gmbh, Vienna, Austria), and concentrations are determined from respective signal intensities according to the Lambert–Beer law.

The ComboSense® is a combined system that integrates an electron impact mass spectrometer (EI-MS) and an ion–molecule reaction mass spectrometer (IMR-MS) within a single analyzer. It employs different ion sources, such as mercury (Hg), xenon (Xe), and krypton (Kr), depending on the analyzed compound, and is calibrated via enhancement calibration to identify interference patterns and linearization models.

Analytical conditions based on tested compounds are detailed in Table S1 in the Supplementary Materials. For measurement, the H_2 gas was directly fed from the Type IV tank system to the FTIR/IMR-EI-MS analysis section using 12 m stainless steel tubing with

SiO_2 coating. Data processing and analysis were carried out using OPUS GA for the FTIR measurements and V&F Client 2.5 for the IMR-EI-MS data. Statistical errors were found to outweigh systematic errors, and overall uncertainty was expressed as three times the standard deviation of the mean value.

3.3. Filter-Based Particulate Matter Assessment (Test C)

During tank defueling, particulate matter was collected via inline filtration. An in-stream filter (FGLP04700—0.22 µm pore size, hydrophobic PTFE, 47 mm membrane, MilliporeSigma (Merck KGaA), Darmstadt, Germany) was pre-conditioned for 24 h in an ISO Class 7 cleanroom environment using a HEPA filter horizontal flow hood [43]. Additionally, the filter holder components were cleaned in an ultrasonic bath of de-ionized water for 20 min, followed by drying in an ISO Class 7 cleanroom environment for 48 h. Then, the pre-conditioned filter was installed between the fueling nozzle and the FCEV receptacle using a hydrogen particle sampler adapter. In alignment with ASTM D7650-21 [44] recommendations, check valves were placed upstream of the tank to prevent reverse gas flow through the filter during defueling.

After defueling, the filter was collected and microscopically assessed using a Zeiss Stemi 305 optical microscope (Carl Zeiss GmbH, Vienna, Austria, 4–200× magnification, 1.25 µm resolution). The particulate load was determined by precision weighing using a Shimadzu AP125WD precision scale (Shimadzu Handelsgesellschaft mbH, Korneuburg, Austria, average of three measurements) in a cleanroom environment.

3.4. Testing and Protocol Completion Criteria

To enable early identification of lubricant formulations with a high risk of degradation or H_2 incompatibility, the testing and protocol completion criteria for the simplified pre-screening protocol are outlined in Table 1. This abbreviated approach comprises only the optical assessment (Test A) and gas analysis aligned with ISO 14687:2025 Grade D (Test B), thereby allowing for rapid screening of prospective lubricant candidates within 24 h. For the full qualification protocol, mandatory testing and protocol completion criteria are summarized in Table 2. Protocol activities, including a defined timeline for the start and end of Test A, Test B, and Test C, are presented in chronological order.

Table 1. Mandatory testing and completion criteria for lubricant pre-screening.

Step	Protocol Activity	Measurement	Related Norm	t = Day 0	t = Day 1
1	Sinter plate preparation	-	-	x	
2	Lubricant application	-	-	x	
3	Gravimetric and optical assessment of lubricant layer	Start of Test A	-	x	
4	Test assembly integration	-	-	x	
5	Refueling	-	SAE J2601 [36]	x	
6	Hydrogen gas analysis	Start of Test B	ISO 14687 [1]	x	
7	Hydrogen gas analysis	End of Test B	ISO 14687 [1]		x
8	Test assembly extraction	-	-		x
9	Gravimetric and optical assessment of lubricant layer	End of Test A	-		x

Table 2. Mandatory testing and completion criteria for full lubricant qualification.

Step	Protocol Activity	Measurement	Related Norm	t = Day 0	t = Day 7–9	t = Day 14–16	t = Day 21–23
1	Sinter plate preparation	-	-	x			
2	Lubricant application	-	-	x			
3	Gravimetric and optical assessment of lubricant layer	Start of Test A	-	x			
4	Test assembly integration	-	-	x			
5	Refueling	-	SAE J2601 [36]	x			
6	Hydrogen gas analysis	Start of Test B	ISO 14687 [1]	x			
7	Hydrogen gas analysis	Monitoring—End of Test B	ISO 14687 [1]		x	x	x
8	PM-filer preparation	-	-				x
9	PM assessment	Test C	ASTM D7650-21 [44]				x
10	Test assembly extraction	-	-				x
11	Gravimetric and optical assessment of lubricant layer	End of test A	-				x

4. Lubricant Suitability Criteria

To assess the contamination potential of lubricants used in high-pressure hydrogen components in FCEVs, the applied methodology included: (i) optical inspection, (ii) gas purity analysis, and (iii) particulate matter analysis. These assessments were selected to detect both short-term and long-term degradation phenomena that could release contaminants into the hydrogen stream and compromise fuel cell system integrity.

First, the optical inspection (Test A) was used to enable elimination of unstable lubricant formulations. Optical stability is considered a key parameter for evaluating physicochemical long-term stability. In particular, in view of the regulatory service life of hydrogen tanks in vehicles of up to 15 years, ensuring stability and sustained functionality is essential [45]. For the pre-screening protocol, the lubricant layer appearance was assessed visually after one day of exposure to high-pressure hydrogen, investigating short-term effects of hydrogen and enabling elimination of unstable lubricant candidates without prolonged testing. In the full lubricant qualification protocol, the optical appearance of the lubricated test assemblies was compared when pristine (day 0) and at the end of the test after extraction from the hydrogen tank in week 4. In both protocols, any form of discoloration, detachment, flaking, spreading, or changes in surface morphology—conditions that could minimize sealing function and lead to persistent tank contamination with the risk of indeterminate release of impurities—was considered unacceptable. Thus, acceptance criteria for the optical inspection were defined as follows:

- Pass: No visible changes in lubricated surface uniformity, adhesion, or appearance.

- Fail: Any visual signs of chemical or physical degradation, such as discoloration, detachment, flaking, spreading, or changes in surface morphology.

The test protocols continue with in-line monitoring of the hydrogen gas purity. In the pre-screening protocol, gas purity was assessed after 1 day of hydrogen exposure. In contrast, for the full qualification protocol, gas purity was evaluated four times up to week 4 to quantitatively ensure that Grade D hydrogen can be delivered to the PEM-fuel cell over representative refill cycles. Gas analysis was conducted in alignment with the ISO 14687:2025 standard, and acceptance criteria were defined as follows:

- Pass: No analyte exceeds ISO 14687:2025 impurity limits at any time point.
- Fail: Exceedance of any ISO-regulated limit.

Then, particulate matter release is assessed only in the full qualification protocol after the total dwell period of the test assembly in the tank. For this, pre-conditioned analytical-grade PTFE filters were placed downstream of the tank outlet to capture entrained solids during controlled depressurization. Filters were weighed and examined using an optical microscope before and after testing to assess both mass change and visual particulate loading. While ISO 14687:2025 does not prescribe a specific particle testing method, this dual-mode approach provides a reproducible means of verifying particulate matter compliance and detecting degradation phenomena not apparent in gas-phase analysis. Acceptance criteria were defined as follows:

- Pass: $\leq$1 mg/kg particulate load in hydrogen.
- Fail: Particulate load above the ISO threshold.

To define the overall protocol outcome, Table 3 summarizes the acceptance criteria and decision logic for the different evaluation scenarios. Specifically, for the pre-screening protocol, a lubricant candidate is classified as potentially suitable if it meets both the optical inspection and gas purity acceptance criteria after one day of hydrogen exposure. In this context, potentially suitable indicates general formulation stability under high-pressure hydrogen during short-term (<24 h) exposure, but does not constitute full qualification for application in FCEV drive trains. A failure in either one or both of the pre-screening tests results in the classification of unsuitable, thereby eliminating the lubricant from further consideration for FCEV drive train applications without additional testing. Similarly, following the full qualification protocol, a lubricant is classified as suitable for use in FCEV drive trains applications only if all three tests (optical inspection, gas purity, and particulate matter) are passed in accordance with the defined acceptance criteria. If the investigated material fails one or more tests, the overall protocol result is designated as unsuitable. Additionally, in the case that a test result is inconclusive while all other criteria have been met, the test must be repeated, as overall protocol completion requires that all individual criteria be satisfied. In contrast, if, for instance, a lubricant fails the optical inspection criterion (Test A), and Test B and/or Test C are inconclusive, the overall outcome is still classified as unsuitable, following a conservative failure assessment approach.

Table 3. Acceptance logic of the staged lubricant qualification framework.

Scenario	Protocol	Optical Inspection (Test A)	Gas Purity (Test B)	Particulate Matter Analysis (Test C)	Total Protocol Result
1	Pre-screening	Pass	Pass	(not tested)	Potentially suitable
2	Pre-screening	Pass	Fail	(not tested)	Unsuitable

Table 3. *Cont.*

Scenario	Protocol	Optical Inspection (Test A)	Gas Purity (Test B)	Particulate Matter Analysis (Test C)	Total Protocol Result
3	Pre-screening	Fail	Pass	(not tested)	Unsuitable
4	Pre-screening	Fail	Inconclusive	(not tested)	Unsuitable
5	Full qualification	Pass	Pass	Pass	Suitable
6	Full qualification	Pass	Pass	Fail	Unsuitable
7	Full qualification	Pass	Fail	Pass	Unsuitable
8	Full qualification	Fail	Pass	Pass	Unsuitable
9	Full qualification	Fail	Inconclusive	Pass	Unsuitable

Collectively, these criteria enable a structured and comprehensive assessment of lubricant suitability, aligned with relevant international standards, including ISO 14687:2025.

5. Results

To evaluate the suitability of lubricants under high-pressure H_2 conditions, the pre-screening and full qualification protocol, including test assembly preparation, insertion into the hydrogen tank, refueling, and subsequent analytic evaluation, were applied as described in Sections 2 and 3 to different lubricants.

First, three formulations with limited information regarding their chemical compositions were provided as semi-masked samples for evaluation in accordance with the pre-screening protocol. The investigation of these semi-masked lubricants was designed to reflect practical scenarios, in which products, protected by confidentiality or trade-secret constraints, must be assessed based solely on the restricted descriptions made available by the manufacturers. Thus, to rapidly evaluate formulation stability and compatibility with high-pressure hydrogen conditions, the simplified pre-screening protocol was applied. The three semi-masked lubricants were assigned internal acronyms and were evaluated based only on supplier-provided composition descriptors and declarations, including a statement of 'no intentionally added PFAS' that was not independently verified by targeted PFAS analysis within the present study. The chemical composition information was provided as follows: (i) SBGL, a silicone base lubricant formulated with aromatic silicone base oil and lithium thickener, chosen for its enhanced oxidation resistance and thermal stability; (ii) MOSS, a high-purity mineral oil thread compound, composed of mineral oil and special solid lubricants, selected for its anti-seize properties and use in threaded connections, such as screws, bolts, and nuts; and (iii) SC, a silicone compound composed of silicone oil and inorganic thickener, which provides a wide service temperature, excellent water resistance, low vapor pressure, and low volatility.

For the full qualification protocol, MOLYKOTE® HP-300 Grease (DuPont Specialty Products GmbH & Co. KG, Wiesbaden, Germany) [46], a fully fluorinated lubricant with PFPE base oil, was supplied by DuPont. It was selected for its low vapor pressure, excellent thermal stability, resistance to chemicals and solvents, and broad compatibility with plastics and elastomers.

In the pre-screening of the three semi-masked formulations, SBGL exhibited a distinct change in the optical appearance of the lubricant layer between the beginning and end of Test A. The initially transparent film became opaque and developed topographical roughness after one day of hydrogen exposure (Figure 4a). These visual alterations potentially indicate degradation processes affecting the molecular structure or surface composition of

the lubricant. Additionally, gas analysis revealed increased concentrations of tert-butyl mercaptan and the halogenated species trichloroethylene, dichloroethane, and the combined fraction of trichloromethane and dichlorobromomethane (Figure 4b–k), supporting the assumption that contaminants were released into the hydrogen stream. Although the total concentration of halogenated compounds remained below the ISO 14687:2025 Grade D limit of 0.05 ppm, the increase of tert-butyl mercaptan warrants particular attention. After 24 h under the high-pressure hydrogen conditions, its concentration reached 0.007 ppm, exceeding the ISO 14687:2025 Grade D threshold. However, the reference measurement indicated that tert-butyl mercaptan was already present at a concentration of 0.004 ppm at the beginning of the test, matching the limit for Grade D purity. Furthermore, the measurement uncertainty at the 24 h time point was larger than 60% of the reported mean value, preventing definitive conclusions regarding regulatory limit exceedance. Thus, for SBGL, Test A was rated a failure, while Test B was considered inconclusive. Accordingly, SBGL was classified as unsuitable based on the optical failure criterion, with the inconclusive gas analysis result not affecting the overall decision outcome.

In the case of MOSS, the black lubricant developed a blistered surface morphology within 24 h of H_2 exposure (Figure 4l). This was accompanied by reduced adhesion to the stainless steel sinter plate, indicating interaction of the lubricant layer and the hydrogen gas in the tank. In contrast, gas analysis revealed no relevant changes in the hydrogen gas purity, with detected contaminants remaining at concentrations well below ISO 14687:2025 Grade D thresholds (Figure 4m–s). These observations suggest that the visual changes are unlikely to result from outgassing of volatile components or chemical degradation, but are more consistent with physical changes to the formulation, such as potential phase separation. Such physical alterations increase the risk of potential particulate matter formation and downstream PEMFC contamination in FCEVs, leading to the disqualification of MOSS based on the optical inspection criterion.

The SC lubricant passed the optical inspection, showing no changes in surface structure and color within the one-day exposure period (Figure 4t). Furthermore, gas analysis revealed only minor variations in contaminant concentrations. Indeed, only a few species, such as hydrocarbons, were detected in the gas stream, none of which increased over the test period or exceeded ISO 14687:2025 limits (Figure 4u–za and Table S2 in the Supplementary Materials), confirming Grade D quality. Thus, SC successfully passed the pre-screening protocol, demonstrating formulation stability under high-pressure conditions over 24 h.

In the full qualification protocol, MOLYKOTE® HP-300 Grease maintained its original surface morphology and visual integrity over the 23-day exposure period, fulfilling the optical inspection criterion according to Section 4 (see Figure 5a). Additionally, gas purity analysis revealed no detectable increase in ISO 14687:2025-regulated impurities over the total dwell period. Indeed, most analytes remained below the limit of detection throughout, and only argon, butane, carbon dioxide, helium, and M29, a mass fragment typical for small organic molecules, were observed after the initial reference measurement (see Figures 5b–f and S2 in the Supplementary Materials). These analytes stayed below ISO 14687:2025 thresholds within the 23-day period, consistently meeting Grade D specification in two separate test runs (see Figure S3 and Tables S3 and S4 in the Supplementary Materials). In the particulate matter assessment, analytical-grade filters placed downstream during controlled defueling showed no visible particles under optical microscopy (Figure 5g) and no increase in mass after filtering 3.1 kg H_2 from the tank. These findings indicate that MOLYKOTE® HP-300 Grease does not release measurable amounts of volatile or solid contaminants during extended exposure to high-pressure hydrogen conditions, passing all acceptance criteria for application in FCEV drive trains.

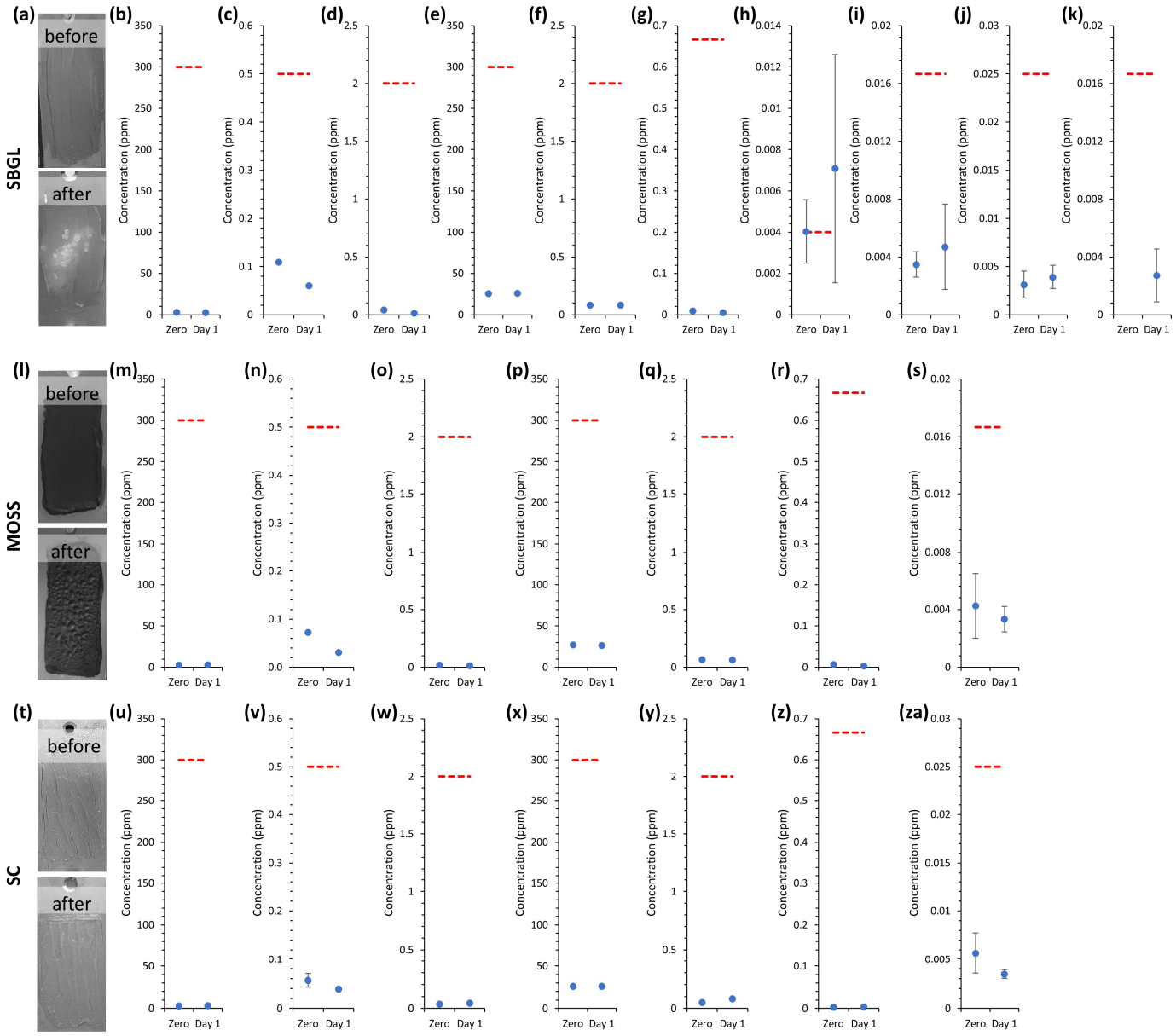


Figure 4. Pre-screening test results, including (**a**) optical inspection of SBGL and (**b**–**k**) gas analysis data of SBGL with (**b**) argon, (**c**) butane, (**d**) carbon dioxide, (**e**) helium, (**f**) M29, (**g**) propylene, (**h**) tert-butyl mercaptan, (**i**) dichloroethane, (**j**) combined fraction of trichloromethane and dichlorobromomethane, and (**k**) trichloroethylene, (**l**) optical inspection of MOSS and (**m**–**s**) gas analysis data of MOSS with (**m**) argon, (**n**) butane, (**o**) carbon dioxide, (**p**) helium, (**q**) M29, (**r**) propylene, and (**s**) combined fraction of trichloromethane and dichlorobromomethane, (**t**) optical inspection of SC and (**u**–**za**) gas analysis data of SC with (**u**) argon, (**v**) butane, (**w**) carbon dioxide, (**x**) helium, (**y**) M29, (**z**) propylene, and (**za**) dichloroethane. ISO 14687:2025 limits are depicted as red dotted lines for Grade D specifications on an element equivalent basis where necessary. Zoomed-in views of the data points, including measurement uncertainties, are provided in Figure S1 in the Supplementary Materials.

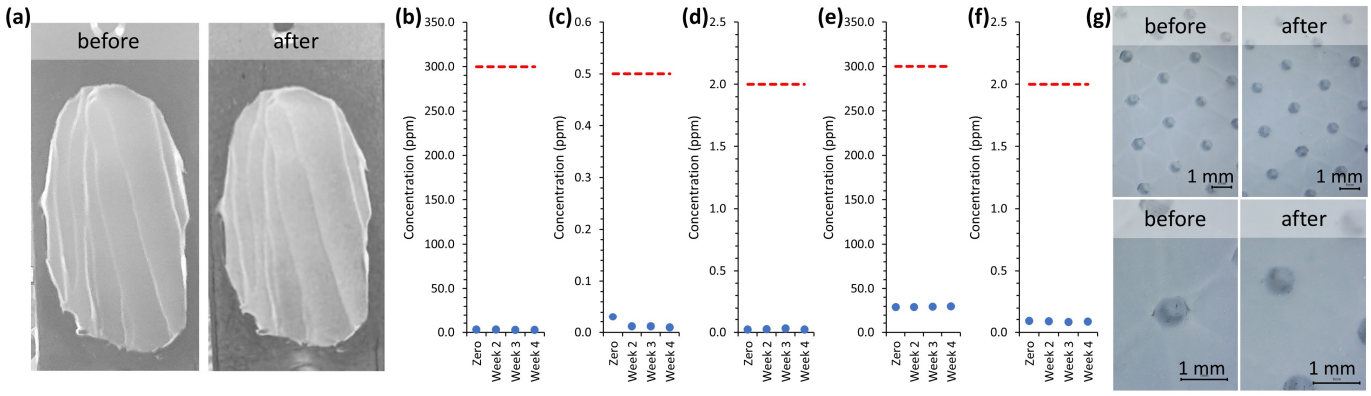


Figure 5. MOLYKOTE® HP-300 Grease results for (**a**) optical inspection before and after exposure of the test assembly to high-pressure H_2 conditions, (**b**–**f**) time-resolved gas purity analysis aligned with ISO 14687:2025 over the 23-day test period, depicting ISO 14687:2025 Grade D limits as red dotted lines and contaminant concentrations as blue markers of (**b**) argon, (**c**) butane, (**d**) carbon dioxide, (**e**) helium, (**f**) M29, and (**g**) optical particulate matter analysis after sampling 3.1 kg of H_2 through a PM filter at the end of the testing period. Zoomed-in views of the data points, including measurement uncertainties, are provided in Figure S2 in the Supplementary Materials.

6. Discussion

6.1. Lubricant-Based Contamination Risks and Implications for Product-Specific Qualification

The findings of this study, summarized in Table 4, indicate that the contamination risk for PEMFC systems is strongly dependent on the specific lubricant product. In the pre-screening trial, which was designed as a rapid gatekeeping step to assess formulation stability and eliminate unsuitable candidates without extended testing, two of the three tested semi-masked formulations did not meet the combined acceptance criteria. Both SBGL and MOSS failed the pre-screening evaluation based on optical inspection, as visible changes in morphology and adhesion behavior were observed. Gas analysis after one day of hydrogen exposure was inconclusive for SBGL and compliant with ISO 14687:2025 Grade D for MOSS, indicating that the observed visual changes cannot be directly associated with volatile contamination above the applied threshold criteria. Since the protocol was not designed to identify degradation pathways or underlying physicochemical mechanisms, the origin of these visible alterations cannot be determined from the present data. Nevertheless, such changes are relevant from an application perspective since flaking, detachment, or particulate formation can contribute to long-term PEMFC damage or impair sealing functionality. As illustrated by these examples, the optical inspection criterion in the pre-screening protocol complements ISO 14687:2025-based gas analysis and provides a simple but effective means of identifying formulation instability and potential contamination-related concerns that may not be captured by gas-quality monitoring alone.

Table 4. Lubricant qualification and pre-screening results.

Lubricant	Testing Protocol	Optical Inspection Result	ISO 14687:2025 Grade D Result	Particulate Matter Result	Total Qualification Result
SBGL	Pre-screening	Fail	Inconclusive	n.a.	Unsuitable
MOSS	Pre-screening	Fail	Pass	n.a.	Unsuitable

Table 4. *Cont.*

Lubricant	Testing Protocol	Optical Inspection Result	ISO 14687:2025 Grade D Result	Particulate Matter Result	Total Qualification Result
SC	Pre-screening	Pass	Pass	n.a.	Potentially suitable
MOLYKOTE® HP-300 Grease	Full qualification	Pass	Pass	Pass	Suitable

n.a.: not applicable.

In the case of SC, both acceptance criteria were met in the pre-screening stage. However, this result should not be interpreted as evidence for silicone-based lubricants as a broader material class. Publicly available information on the hydrogen compatibility of individual lubricant products remains limited, and only a small number of in situ fuel cell studies are available. While voltage losses have been reported for certain silicone-containing sealing materials during in situ testing [28], the differing outcomes observed for SBGL, SC, and the materials reported in the literature highlight that contamination risk cannot be inferred reliably from the nominal base material or material class stated in manufacturer descriptions alone. In particular, the possible influence of undisclosed additives, impurities, or formulation-specific processing effects cannot be assessed without more detailed compositional information, which supports the need for product-specific qualification for the use in high-pressure hydrogen FCEV tanks.

For the full qualification protocol, the results obtained for the MOLYKOTE® HP-300 Grease show that this specific PFPE-based product met the acceptance criteria under the investigated conditions. Literature on fluorinated materials reports properties such as low vapor pressure, chemical inertness, and thermal stability [47], which may provide useful context for interpreting this favorable outcome. In addition, similar fluorinated materials showed no significant impact on fuel cell performance during several hours of in situ exposure testing [28], and several fluorinated products are described by manufacturers as suitable for hydrogen applications (Table S5 in the Supplementary Materials). However, these observations should be regarded as contextual support rather than direct evidence for fluorinated lubricants as a class in the present application. Both the number of fluorinated products assessed in this study and the amount of publicly available in situ evidence remain limited. Moreover, detailed compositional data, characterization results, and qualification criteria for high-pressure hydrogen applications are rarely disclosed by manufacturers. Accordingly, no class-level conclusion can be drawn for fluorinated lubricants in general. This is particularly relevant considering that ISO 14687:2025 Grade D specifies impurity limits as low as 4 ppb, highlighting that even trace levels of contaminants could impact hydrogen quality. Consequently, it is emphasized that contamination risk cannot be assessed solely on the basis of the reported formulation type, and lubricant qualification on a product-specific basis is considered essential.

6.2. Protocol Applicability, Advantages and Constraints

While in situ FC testing is planned for the lubricants tested in this study to allow detailed comparison with other materials using the same methodology [48], the advantages and limitations of the protocol presented here warrant discussion. In situ testing directly measures the time-resolved influence of materials on FC performance, but the setup is complex, requiring multiple components, sensors, controls, and often specialized operational expertise. In contrast, the protocols presented here do not assess impact on FC performance directly. Instead, they evaluate lubricants for refueling of high-pressure hydrogen tanks

using a simple and conservative analytical workflow, combining optical and gravimetric assessment with hydrogen gas purity evaluation aligned with ISO 14687:2025 Grade D, and, for full qualification, additional filter-based particulate matter analysis. Moreover, the complexity and knowledge requirements of the presented method are less demanding compared to in situ testing setups. The approach relies on a commercially available Type IV tank and standardized SAE J2601 refueling profiles, which reproduces the relevant exposure under realistic conditions and targets post-dispensing contamination along the same gas pathway that would feed an FC system. In the present implementation, gas purity was monitored in-line. However, the protocol can in principle also be executed using external gas analysis facilities provided that appropriate sampling procedures are applied, which may further lower the barrier for adoption. A practical limitation is throughput: only one lubricant can be assessed per tank campaign, and small-scale tests were intentionally avoided to preserve realistic conditions. Nevertheless, operator workload is low since intervention is required mainly at the start and end of a test and at defined intermediate analysis time points, while the system can remain unattended during dwell periods.

With respect to robustness and reproducibility, the testing framework is anchored in internationally recognized standards for both exposure and acceptance criteria, providing a structured basis for lubricant assessment under realistic refueling conditions. However, the broader generalizability of the protocol must be interpreted in the context of the presented validation scope: three lubricant formulations were assessed in the 24 h pre-screening stage, while full qualification was completed for only one lubricant. Accordingly, the current dataset primarily demonstrates feasibility and decision logic rather than establishing cross-comparability across lubricant products and classes. Nonetheless, the examples presented in this work demonstrate that the pre-screening stage is suitable for identifying formulation stability and potential contamination risk within 24 h, and full qualification outcomes are reproducible across independent test runs (see Tables S3 and S4 in the Supplementary Materials). However, limited knowledge can be gained with respect to the observed formulation changes and contaminant release, as the testing framework is not intended to elucidate underlying degradation mechanisms. It is also noted that statistically robust conclusions will require expanded material sets and a larger number of replicates.

Beyond mobile applications, the authors note that the methodological logic of this work may also be transferable to stationary hydrogen systems in the future. This transferability is not validated in the present work and will require dedicated testing. However, boundary conditions are similar for both applications. The laboratory setup, which fulfills hydrogen purity requirements equivalent to ISO 14687:2025 Grade D, is likewise capable of meeting more stringent Grade E specifications associated with stationary fuel cell applications [1]. In such systems, particularly those employing steel tanks, similar physicochemical phenomena are expected. Unlike composite Type IV tanks in FCEVs, stationary steel vessels are regulated under the Pressure Equipment Directive rather than SAE refueling temperature-management protocols. Consequently, higher fill temperatures can be permissible. This results in locally elevated temperatures during filling, potentially intensifying lubricant volatilization and degradation processes, which could be evaluated based on the optical and gas purity tests. Furthermore, stationary installations often comprise extended piping networks and steel fittings, which might contribute to an increased particle load originating from welding and manufacturing residues. The inclusion of particulate matter assessment in the transferred full qualification pathway would therefore be particularly relevant for such scenarios and could be extended to the pre-screening stage without changing the overall workflow.

Combined, the pre-screening and full qualification protocols provide a staged framework to identify unstable lubricant formulations early and to support subsequent full qualification of selected candidates under application-relevant conditions.

7. Conclusions

Testing protocols for evaluating lubricant-derived contamination in high-pressure hydrogen systems were developed. A simplified pre-screening protocol was defined based on optical inspection and gas purity requirements, enabling rapid evaluation and potential candidate elimination based on lubricant formulation stability within 24 h. Additionally, the full qualification approach integrates optical inspection, in-line gas purity monitoring over 23 days, and particulate matter analysis following realistic refueling events. By carefully controlling test conditions and adhering to established international standards for refueling and gas quality, the protocols provide a structured and conservative method to qualify lubricants for use in FCEV tanks at different evidence levels.

The staged framework was applied to four lubricants to demonstrate feasibility of the decision logic under realistic 700 bar refueling exposure. Three candidates were assessed via pre-screening: one silicone-based formulation met the 24 h acceptance criteria and was classified as potentially suitable, whereas two other lubricants were classified as unsuitable due to formulation instability. In the full qualification protocol, the PFPE-based MOLYKOTE® HP-300 Grease met all acceptance criteria over 23 days in two separate test runs, resulting in the classification of suitable. The observed product-specific differences show that lubricant compatibility cannot be inferred reliably from nominal material class or supplier descriptions alone. Instead, application-relevant and product-specific qualification is required. The proposed staged framework therefore provides a practical pre-normative basis for material selection, supplier qualification, inter-laboratory validation, and future standardization of compatibility testing for lubricants in FCEV hydrogen tanks. In this way, the protocol supports both safer integration of lubricants into high-pressure hydrogen tank systems and the long-term reliability of PEMFC-based mobility applications.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/hydrogen7020083/s1, Figure S1: (a–j) Gas analysis data of SBGL with (a) argon, (b) butane, (c) carbon dioxide, (d) helium, (e) M29, (f) propylene, (g) tert-butyl mercaptan, (h) dichloroethane, (i) combined fraction of trichloromethane and dichlorobromomethane and (j) trichloroethylene; (k–q) gas analysis data of MOSS with (k) argon, (l) butane, (m) carbon dioxide, (n) helium, (o) M29, (p) propylene and (q) combined fraction of trichloromethane and dichlorobromomethane; (r–x) gas analysis data of SC with (r) argon, (s) butane, (t) carbon dioxide, (u) helium, (v) M29, (w) propylene and (x) dichloroethane; Figure S2: Gas analysis results of DuPont™ MOLYKOTE® HP-300 Grease, showing contaminant concentrations with error bars of (a) argon, (b) butane, (c) carbon dioxide, (d) helium, (e) M29; Figure S3: Gas analysis results of DuPont ™ MOLYKOTE® HP-300 Grease (2nd run) with ISO 14687:2025 limits presented in red lines and contaminant concentrations of (a) argon, (b) butane, (c) carbon dioxide, (d) helium, (e) M29, (f) propylene, (g) combined fraction of trichloromethane and dichlorobromomethane; (h–n) contaminant concentrations with error bars of (h) argon, (i) butane, (j) carbon dioxide, (k) helium, (l) M29, (m) propylene, (n) combined fraction of trichloromethane and dichlorobromomethane; Table S1: Overview of Equipment used for ISO 14687 contaminant analysis; Table S2: Hydrogen quality analysis result of SC lubricant, showing H2 quality Grade D: 4.5 (99.997%)* after 1 day of exposure in a 700 bar Type IV vehicle tank; Table S3: Hydrogen quality analysis result of MOLYKOTE® HP-300 Grease (run 1), showing H2 quality Grade D: 4.5 (99.997%)* within 23 days of exposure in a 700 bar Type IV vehicle tank; Table S4: Hydrogen quality analysis result of MOLYKOTE® HP-300 Grease (run 2), showing H2 quality Grade D: 4.5 (99.997%)* within 23 days of exposure in a 700 bar

Type IV vehicle tank; Table S5: Fluorinated lubricants classified for hydrogen application. Ref. [28] is added in the Supplementary Materials.

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Abbreviations

The following abbreviations are used in this manuscript:

FCEV	Fuel cell electric vehicle
PEM	Proton exchange membrane
PEMFC	Proton exchange membrane fuel cells
OTV	On-tank valve

NRL	National Laboratory of the Rockies
FTIR	Fourier transform infrared
IMR-MS	Ion–molecule reaction mass spectrometer
EI-MS	Electron impact mass spectrometer

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