Publishable Summary for 19ENG04 MetroHyVe 2
Metrology for hydrogen vehicles 2

Overview

To meet the 2050 Europe carbon neutral targets many countries are now legislating to ban the sale of internal combustion engines. Electric vehicles, running on either hydrogen fuel cells (FC) or rechargeable batteries, are a viable alternative. Specifically, the automotive industry considers FCs to be a real breakthrough for e-mobility and could represent 32 % of fuel demand by 2050. Current barriers to mass implementation of hydrogen in transport arise from European Directive 2014/94/EU on the deployment of alternative fuels infrastructure and International Organisation of Legal Metrology (OIML) recommendations. This project has addressed these issues by developing the necessary infrastructure and tackling measurement challenges in hydrogen flow metering, quality control and sampling as well as fuel cell stack testing. Following project achievements, Hydrogen refuelling stations flow meters can be calibrated and verified by 4 primary standards in Europe, more than 13 laboratories have demonstrated their ability to perform hydrogen fuel analysis, new reference materials and calibrants are available for analytical laboratories enabling full calibration and verification of hydrogen fuel quality, 4 sampling systems have been compared and new guidance for reliable fuel cell testing is available for the industry to improve best practices, technical evidences supported the first hydrogen fuel sampling standard development at ISO level. This will ultimately enable hydrogen to become a conventional fuel and support the European energy transition.

Need

Climate change, air quality and reliance on imported fuels from non-renewable sources require quick deployment of alternative fuels such as hydrogen to meet the 2050 Europe carbon neutral targets (net-zero emission from transport). The previous EMPIR project 16ENG01 MetroHyVe addressed hydrogen for transport measurement challenges and has established a solid basis of research for this project to build upon. Through engagement with key stakeholders such as consumers, hydrogen producers, HRS operators, automotive manufacturers and standardisation bodies, the previous project identified the following measurement challenges that need to be addressed in order to support the deployment of hydrogen as a transport fuel in Europe:

- Consumers need confidence that they are being charged correctly when buying hydrogen at a HRS. It was previously not possible to accurately measure the amount of hydrogen dispensed when filling hydrogen into a heavy-duty FCEV. (Objective 1)
- HRS compliance to regulation is currently expensive to achieve and to maintain. To reduce the subsequent costs to the consumer, HRS operators need the ability to test their stations in a quicker, cheaper and more frequent manner. Methods using secondary standards for hydrogen flow dispensed at HRS calibrated by the European primary standards were urgently needed for both light and heavy-duty vehicles. (Objective 1)
- To improve reliability of their results and hence facilitate improved confidence of end users, commercial gas analysis laboratories in Europe needed hydrogen fuel reference materials for the measurement of hydrogen fuel quality according to ISO 14687:2019. (Objective 2)
- There were no harmonised guidelines or ISO standards for hydrogen fuel sampling at HRS. Sampling bias due to improper strategy, materials, purging or delay for analysis may have led to biased information on hydrogen fuel quality. There was a high risk that the results received by consumers were not representative of the actual hydrogen dispensed into the FCEVs due to improper sampling. Standardisation bodies (ISO TC 197) needed technical evidence to revise the standards (ISO 19880-1). (Objective 3)
- HRSs operators needed to install sensors that can continuously monitor key contaminants to ensure that such contaminants never reach the FC vehicles; some instruments have been tested in laboratories during the previous EMPIR project 16ENG01 but the sensors and analyser were never validated in real life conditions at HRS. (Objective 4)

- A better understanding of the impact of contaminants on stacks was needed by automotive manufacturers, tier 1 suppliers, research institutes, hydrogen producers and normalisation committees to ensure the lifetime of FCEV is optimised. Studies based on using hydrogen recirculation loop produce more reliable data on the effect of contaminants on the FC stack. However, there was no harmonised methodology which may have led to unreproducible results or biased interpretation. As the current ISO 14687:2019 (on Hydrogen fuel quality — Product specification) threshold values are based on such previous studies, it was vital to harmonise this methodology. (Objective 4)

Additionally, there were no recommendations or good practice guides for European regulation bodies for the verification of HRS with respect to handling, duration and long-term behaviour.

Objectives

The overall aim of the project was to develop a broad underpinning metrological infrastructure to support the European hydrogen industry.

The specific objectives of the project were:

1. To develop a metrological framework for testing hydrogen meters used to measure the mass of hydrogen dispensed into light-duty to heavy-duty fuel cell electrical vehicle (FCEV) from HRS, traceability was assured by developing new primary standards (< 0.5 % accuracy) or secondary standards (< 2 % accuracy) traceable to the primary standards of previous EMPIR project 16ENG01 MetroHyVe. Guidelines and a good practice guide to ensure accurate measurements and minimised uncertainty due to hydrogen refuelling stations (HRS) design was produced.
2. To develop reference materials (RM) suitable to monitor hydrogen quality laboratory performance and to undertake an inter-laboratory comparison on hydrogen fuel including all contaminants regulated in EN 17124:2018 and ISO 14687:2019. Guidelines for online analysers at HRS over long periods (including validation, calibration and quality control) and recommendations to ISO 21087:2019 for online analysers and sensors were produced.
3. To develop and validate sampling equipment and procedures based on European and national projects' feedback as well to harmonize proposals for hydrogen sampling methods with USA and Japan. Good practice guides on hydrogen sampling at HRSs (nozzle) and other locations (e.g. FCEV) to provide input for the revision of ISO 19880-1 were also produced.
4. To develop standard test protocols, including reproducibility study and intercomparison evaluation for automotive fuel cell (FC) stack testing (with online analysis) to determine threshold limits of critical contaminants in hydrogen. Critical parameters for FC stack testing with anode recirculation loop were defined. A good practice guide on the measurement of impact of contaminant on FC stack (with online analysis) with detailed description of parameters to monitor was developed.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project to the hydrogen industry including the measurement supply chain (such as hydrogen quality laboratories, instrument manufacturers), standards developing organisations (ISO/TC 197, CEN/CENELEC and IEC TC105) and end users (hydrogen fuelling stations, vehicle manufacturers).

Progress beyond the state of the art

Objective 1 - Flow metering: Currently there are more than four primary standards installations in Europe (including METAS, CESAME, NEL and JV) for calibrating hydrogen flow meters to be used at HRSs for light-duty vehicles. METAS and Cesame have demonstrated calibration procedures for monitoring the amount of hydrogen dispensed into the vehicle up to pressures of 875 bar required for stations providing hydrogen at nominal working pressure (NWP) of 700 bar. The current primary standards are compliant for fuel delivery to light-duty vehicles according to the Society of Automotive Engineers (SAE) J2601 protocol but an international field comparison for clear acceptance by the HRS operators and notified bodies was missing. The partners realised the first international field comparison at ZBT test field HRS and are in the process of calibration and measurement capabilities claims. Implementation of heavy-duty vehicles such as buses and trucks into the hydrogen infrastructure requires primary standards that can go up to higher flow rates and therefore require a dedicated standard, which is now available thanks to the project at METAS and NEL. The project went beyond the state of the art by developing a gravimetric flow method to calibrate and verify flow meters with hydrogen that are compatible with heavy-duty vehicles. The project went beyond the state of the art by developing and demonstrating the traceability chain from primary standards to secondary standards for regular verification of small and light-duty vehicles as well as for heavy-duty vehicles. Additionally, regular monitoring of HRS with different designs using the already available gravimetric standards from the previous EMPIR project 16ENG01 MetroHyVe allowed to provide recommendations for the periodicity of verification.

Objective 2 - Hydrogen quality control including inter-laboratory comparison including all contaminants: European laboratories are developing the capability to measure the contaminants specified in ISO 14687:2019 and EN 17124:2018. However, the current lack of gas calibrants with sufficient uncertainty, reference materials (RM) for validation and inter-laboratory comparison scheme were a barrier for European commercial laboratories to be able to prove their agreement to ISO 21087:2019. This project went beyond the state of the art by developing potentially dozens of gas calibrants and the RMs (composition and gas cylinders) that European laboratories need to validate their analytical methods according to ISO 21087:2019. In the previous project 16ENG01 MetroHyVe a hydrogen quality inter-comparison was performed with 4 of the contaminants specified in ISO 14687:2019. This project went beyond state of the art by completing a new inter-comparison involving 8 contaminants in ISO 14687:2019. The previous EMPIR project 16ENG01 MetroHyVe validated 5 online gas analysers in laboratory-based conditions. However, such online hydrogen analysers and sensors have yet to be validated and tested at HRSs to ensure their performance meets the criteria for implementing hydrogen quality control strategies as suggested in ISO 19880-8. This project went beyond state of the art by testing and validating online hydrogen purity sensor in laboratory condition and analysers and sensors at HRSs. It provided metrological guidance to support accuracy and traceability of such measurements.

Objective 3 - Sampling: Representative sampling of hydrogen is critical and currently some contaminants such as H_2S or water are hidden or enhanced by inappropriate sampling strategies. The previous EMPIR project 16ENG01 MetroHyVe started investigating the stability of some contaminants, such as CO or H_2S in commercially available sampling vessels. However, the number of sampling cylinders and additional contaminants such as CH_2O, CH_2O_2 or NH_3 requires additional studies. This project performed robust tests to evaluate reactive compounds' stability in commercially available sampling vessels and in state-of-the-art sampling vessels (over 9 types of cylinder tested for 5 contaminants). The sampling vessel is only a part of the sampling strategy and three sampling methods at an HRS nozzle have been recently reported in ISO 19880-1 annex K. However, the sampling methods were not validated or compared, which has raised concerns on sampling equivalence worldwide. The project went beyond the state of the art by performing the first sampling inter-comparison of all the European hydrogen sampling strategies at the HRS nozzle (> 5 sampling systems tested including US and Japanese approaches).

Objective 4 - FC stack testing: Harmonisation of automotive FC stack testing is needed for the assessment of their performance and durability under operating conditions (including reproducibility issues) and hence of their technological progress. The project went beyond that state of the art by providing the first guideline for FC stack testing with a recirculation loop. The project tested the reproducibility of FC stack testing and highlighted the critical parameters for their performance, as well as harmonising the test protocol.

Results

Objective 1 – Development of a metrological framework for testing hydrogen meters used to measure the mass of hydrogen dispensed into light-duty to heavy-duty fuel cell electrical vehicle (FCEV) from HRS

The consortium completed the development of two first primary standards for heavy-duty vehicle (35 and 70 MPa) with completion in spring 2023. The uncertainty limit was very challenging due to the ratio 'mass to be determined' vs 'mass to be weighed' that puts hard constraints on the accuracy of the weighing system, as well as the important impact of buoyancy correction on the uncertainty budget. The key comparison of flow metering primary standards for light duty vehicles refuelling stations was completed at ZBT test field by the partners. The field measurements campaign was completed to gather data on stability of the meter and establish verification periods and recommendations were made. The use of a secondary standard was developed and validated by field-measurements. An uncertainty model for HRS dispensers to be used by HRS designers and notified bodies to better understand uncertainty sources has been developed and matches real values well.

Objective 2 – Develop reference materials (RM) suitable to monitor hydrogen quality laboratory performance

The partners completed the 1-year stability testing of the following reference materials:

- Three new multi-contaminant gas reference materials in hydrogen in a large variety of gas cylinders. The mixtures cover contaminants found in real hydrogen fuel samples and detailed in ISO 14687.
- The partners completed, the 1-year stability study for formaldehyde and ammonia in hydrogen matrix in different gas cylinders and preparation to develop new reference materials for calibration suitable for hydrogen purity laboratory.

Results from the stability study showed the best cylinder or composition to achieve low uncertainty (< 20%) and at least 1 year stability.

Based on the reference materials development, the interlaboratory scheme covering eight contaminants (specified in ISO 14687) in hydrogen fuel was feasible, it is the most ambitious interlaboratory comparison worldwide. The partners produced a batch of complex gas mixtures (14 gas mixtures), largest number of contaminants in gas cylinder (8 compounds) including reactive compounds at ISO 14687 threshold (i.e., 4 nmol/mol of sulphur compounds). The 13 laboratories (including commercial laboratories) from Europe and North America received their cylinders and realised their measurements. The results were evaluated and reported. Corrective actions were implemented enhancing the performance of some participants.

Objective 2 - Guidelines for online analysers at HRS over long periods (few days to 1 month)

The online analysis at three refuelling stations was realised using 4 different sensor types and 8 different online analysers for few days and up to month in operation. The results highlighted the lack of ATEX analysers, the complexity to implement online analysers at HRS, the need for calibration or verification onsite, importance of analysis frequency, limit of detection, response time and delay. Guidelines were developed based on the results of field experience.

Objective 3 - Development and validation of sampling equipment and procedures

The study of reactive compounds' behaviour in all the different sampling cylinder types was completed (4 studies of up to 5 compounds in 9 types of cylinder). The partners achieved a bilateral sampling comparison between the European and US approaches (in collaboration with ASTM) which showed good agreement. The sampling inter-comparison in Europe was realised by the partners with four different sampling systems in October 2022. It highlighted the impact of the hydrogen refuelling station parameters on the representativeness of the sampling. A repetition was realised in September 2023 with more control of the refuelling station parameters and achieved good agreement between four systems. The intercomparison between Asia and Europe was completed too. The partners have updated the 16ENG01 MetroHyVe good practice guide for representative sampling and analysis of particulate matter in hydrogen fuel dispensed from HRS (available on MetroHyVe 2 website). A peer review article on particulate sampling was published based on the partner's research. A new infrastructure to test the contaminant behaviour in FCEV tanks and FCEV has been developed and tested by project partners. The experiments were performed on real tanks and FCEV to evaluate the behaviour of reactive and non-reactive contaminants in hydrogen along the vehicle's key components. As a new methodology for sampling contaminants, sorbent tubes were successfully tested at high pressure (few bar) for capturing and transporting very low-level reactive impurities (available on MetroHyVe 2 website).

Objective 4 - Development of standard test protocols, including reproducibility study and intercomparison evaluation for automotive fuel cell (FC) stack testing

The consortium finalised an extensive literature review and first comparison to evaluate their state-of-the-art systems. The partners performed a metrological evaluation of the degree of equivalence of four test benches on the fuel cell performance results. The results were key to develop a good practice guide on measuring the impact of contaminants on fuel cell stacks (available on MetroHyVe 2 website).It will significantly improve equivalence between laboratories and fuel cell results in Europe and worldwide and help better benchmark of fuel cell performance for the development of large scale production of fuel cell or improvement of performance for heavy duty application. The test protocol reproducibility study was completed between three partners using well known contaminant (CO) including oxygen online monitoring. Comparison of more than 4 different online analysers was realised by the partners in recirculation loop. The studies on clean-up protocol of fuel cell pipes after exposure to contaminants (inert, hydrocarbons, reactive species) in hydrogen were completed and were essential to define good practice for this complicated study.

This project successfully met all objectives.

Impact

The key dissemination activities of the project were its contribution to standards developing organisations with participation in meetings for ISO TC 197 WG 24, 27 and 28, and CEN TC268 and CEN/CLC JTC6. The partners supported the revision of ISO 14687 and ISO 19880-8 providing technical input and the drafting of ISO 19880-9 for hydrogen sampling standardisation (convened by partners in this project). In addition, a stakeholder workshop for flow metering was held virtually and another workshop for flow sampling was held at Air Liquide in France. Over 200 experts from industry, research and policy makers attended the events and were informed about the achievements of the project.

The project partners have also presented the importance and relevance of hydrogen fuel quality measurements during European (Gas conference 2022, EHEC) and international conferences (World hydrogen energy conference, world hydrogen technical conference, Hypothesis, IMEKO). Technical reports and good practice guides have been freely available for the Hydrogen community. Moreover, the project emulated novel research activities in related fields (i.e., heat transfer in hydrogen tank published article by partners).

Impact on industrial and other user communities

The project produced validated primary standards for type-approval and initial verification of HRS as well as a new traceability chain for secondary standards. These have been made available to HRS operators and should allow quick and affordable verification of HRS for both light- and heavy-duty FCEVs. HRS operators have already begun to use primary standards from partners which has resulted in verification of LD and HD HRS according to OIML R139-1 in Europe. Additionally, new measurement capabilities to allow HRSs to charge customers correctly when refuelling heavy-duty FCEV will be made available because of the project. (Objective 1). HRS operators and notified bodies will have access to several internationally recognised primary standards for initial verification and type-approval as well as traceable secondary standards for flow verification according to OIML recommendation. Considering the deployment of 4500 HRS in Europe by 2030 compliant to local and European regulations, the project outcomes will support significant reduction in the cost and time of flow verification while ensuring accuracy and traceability.

More than five commercial gas analysis laboratories and instrument providers joined the intercomparison (13 participants), the results showed relatively good performance and highlighted the points to improve. The results from this intercomparison have assisted the participants (such as commercial analytical laboratory) to support claims on their ISO 14687:2019 capabilities and competence in compliance with ISO 21087:2019. This will enable them to provide reliable results to HRS operators and to claim ISO 17025 accreditation for hydrogen fuel analysis. The novel reference materials developed in the project are already being taken up by commercial gas analysis laboratories and instrument providers. This will allow them to demonstrate their competence for hydrogen fuel quality measurement. (Objective 2). The deployment of the accurate and traceable European hydrogen quality laboratories will help to prevent serious damage to FCEVs which would be costly for the automotive manufacturers and fleet operators. Considering the European target of 1 million FCEVs in 2030, the reduction in risk of FCEV damage due to low quality hydrogen fuel is a critical cost saving for fleet operators (estimated to be millions of euros).

HRS operators will benefit from the consortium reports to install and maintain accurate online hydrogen analysers and sensors. Therefore, HRSs will be able to obtain reliable hydrogen fuel quality analyses using external laboratories or online measurements in compliance with ISO 14687:2019 and ISO 19880-8 following EU Directive 2014/94/EU. (Objective 2). The implementation of quality control using online hydrogen purity analysers and sensors reduces the need for subcontracting regular hydrogen purity analysis by a commercial laboratory. It will result in lowering the price of hydrogen compared to conventional fuel and will be beneficial to the end users, FCEV owners and the automotive manufacturers.

The state-of-the-art peer-review article (https://doi.org/10.1016/j.ijhydene.2021.08.043) including suitable sampling vessels, suitable equipment, procedures, and delay for analysis can be followed by HRS operators and applied by analytical laboratories. It will support confidence of end-users and ensure optimised lifetime of FCEVs. Bilateral comparison within EU, between EU and ASTM has been realised. (Objective 3). The development of FCEV sampling was extremely useful to the industry to test hydrogen quality directly from buses and understand hydrogen fuel quality issue that could have been detrimental to their fleet.

Harmonised stack testing will support the FC industry, automotive manufacturers and suppliers in developing improved stacks in terms of their lifetime or the impact of contamination on them. The project's results will also help to potentially identify new types of stacks for the next FCEV generation. (Objective 4).

Through the support into FCEV reliability, HRS fuel quality and cost reduction, the FCEVs will become more visible to society. This will enhance acceptance of FCEVs as “normal” road vehicles rather than a prototype. The societal acceptance of hydrogen fuel is essential to achieve the energy transition toward a greener society. In addition to supporting climate change targets, introducing taxi fleets, hydrogen buses and heavy-duty vehicles to market will improve cities' air quality and provide health benefits as only water is emitted at the tailpipes; this will prevent people from breathing in toxic emissions and reduce the frequency of pollution peak.

Impact on the metrology and scientific communities

Primary standards to verify hydrogen flow meters under real conditions (i.e., 875 bar) will be made available by the project for a large spectrum of vehicles (light and heavy-duty) and high flow stations. Data sets for the establishment of recommendations for the periodicity of verification of HRS based on their design was developed by the project. NMIs and DIs, using the primary standards developed in this project will be able to provide traceability to industry secondary standards for flow verification and claim CMCs. Considering the target of 4000 HRS in EU by 2030, the use of primary standard to perform HRS verification is impossible to achieve. The secondary standards developed in this project will enable existing flow calibration laboratories to start providing a calibration service for the secondary standards needed by hydrogen industry enabling the requested number of verifications for European and local requirements.

New reference materials, primary gas mixtures and certified gas mixtures will be made available to analytical laboratories, sensor, instrument manufacturers to develop the quality control chain for the hydrogen fuel industry (HRS, automotive manufacturers).

Electrochemistry laboratories and automotive industry will be able to perform reproducible stack test with recirculation loop using harmonised protocol. It will allow comparison of results obtained in Europe and between institutes and industry and potentially support quicker identification of the new type of stack for the next FCEV generation.

The project promoted international collaboration and comparison on hydrogen quality (sampling and analysis between Europe, Asia and US) to support the global hydrogen economy.

Impact on relevant standards

The project will support the revision of international recommendation on compressed gaseous fuel metering measuring systems for vehicles, OIML R139, with technical evidence and guidelines on calibration, achievable uncertainty, flow metering for light and heavy-duty vehicles at HRS and verification periodicity.

The consortium peer-review articles and results were shared with experts from international standardisation committees working on ISO 14687, ISO 19880-8 and on the future ISO 19880-9 standard. It supported the drafting of new sampling standards for hydrogen at HRS nozzle ISO 19880-9 (ISO/TC 197/WG 33).

The harmonised test protocol on the reproducibility of FC stack as well as the recommendations and good practice guide will be provided to ISO/TC 197/WG27 to revise the standard ISO 14687 on hydrogen fuel contaminant limits. Moreover, as the ISO 14687:2019 threshold values are based on such studies, reproducibility and harmonised test protocol will support the WG in revising the standard with reliable technical results. This work will be valuable to support the revision of IEC 62282-7-1 for FC stack testing with anode gas recirculation within IEC TC105. Proposal for guidelines on online analyser and sensor validation and quality control at HRS will be provided to the ISO TC197 WG28 and ISO TC 158 JWG7 (for revision of ISO 21087:2019 and ISO 19880-8). The project will support CEN/CLC/TC 6's decision whether ISO 19880-9 can be adopted at CEN level. The project provided reports on hydrogen contaminants measurements, quality control of offline laboratory and online sensor and analyser, sampling strategies and recommendations to CEN/TC 268/WG 5 to support the revision EN 17124:2018. Finally, this project supported the revision of IEC 62282-7-1 for FC stack testing with anode gas recirculation.

Longer-term economic, social and environmental impacts

Replacing conventional petrol and diesel vehicles will help Europe reach the challenging emission targets. Deployment of hydrogen for transport will support the growth in hydrogen production using renewable energy such as solar or wind to decrease the entire emission footprint of the sector. The emissions from hydrogen vehicles (i.e., buses, vans, trucks, taxis) are safe for health and will help reduce urban pollution and pollution peaks responsible of hundreds of thousands of deaths per year.

The standards and methods developed in this project will ensure that FCEV and heavy-duty vehicles can go to market without hindrance from the technical specifications in European Directive 2014/94/EU. A quicker deployment of FCEV fleets will help to reduce carbon dioxide emitted from cars, and especially freight carrying vehicles, which is crucial for reaching the very challenging climate change targets set at the Paris Climate Conference (COP21) and the new European commission target for 2050.

List of publications

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[3] *Heat transfer analysis of high pressure hydrogen tank fillings,* Couteau, A., Eggenschwiler, P.A. and Jenny, P., International Journal of Hydrogen Energy 47 (2022) 23060-23069.
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[4] *Review of sampling and analysis of particulate matter in hydrogen fuel*, Aarhaug, T.A., Bacquart. T., Boyd, R., Daniels, C.International Journal of Hydrogen Energy, 49 (2023), 1293-1305,
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[5] *Secondary standard for hydrogen refuelling station verification: Method and requirements*, Maury, R., De Huu, M.A., MacDonald, M., Neuvonen, P. T., Wiener A., Proceeding 19th International Flow Measurement Conference 2022,
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[8] *Assessing the Performance of Fuel Cell Electric Vehicles Using Synthetic Hydrogen Fuel.* Bacquart, T.; Storms, W.; Moore, N.; Olden, J.; Morris, A.S.O.; Hookham, M.; Murugan, A.; Mattelaer, V. Energies 2024, 17, 1510.
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[9] *An inter-laboratory comparison between 13 international laboratories for eight components relevant for hydrogen fuel quality assessment*. Arrhenius, K., Morris, A., Hookham, M., Moore, N., Modugno, P., Bacquart, T. Measurement 202
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This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

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