



Sealant Suitability

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Executive summary

This study aimed to determine whether sealants commonly used in the Natural Gas industry are compatible with hydrogen. A test programme was carried out on a range of sealants selected with consultation from industry experts. The sealants tested generally comply with BS EN 751 section 1 (Anaerobic Sealants), section 2 (Non-hardening Jointing Compounds), or section 3 (Unsintered PTFE Tapes and Strings). The scope of this project is downstream domestic and industrial and commercial installations, up to 75 mbar maximum operating pressure (MOP).

Throughout this report, particularly in the results section, there is reference to 'support material' for the compounds. This refers to PTFE cord, PTFE tape, or hemp fibres. This is generally a fibrous substrate that is more solid than the compound and works together with the compound to produce a robust seal with very low residual leakage.

Accelerated ageing was done using the Arrhenius temperature method to determine whether chemical reactions between the sealants and hydrogen caused any increase in leakage. The artificial ageing test programme lasted 41 weeks, with one of each test piece at room temperature (20 °C), one at 60 °C to represent a maximum of 13 years, and one at 80 °C to provide a 50 year comparison.

The leak rates are given in ml/min as this is appropriate for the level of leakage and precision used. The summary of results also presents these leak rates in m³/hr, to provide context within the gas industry. All leak tests were carried out using hydrogen at 150 mbar (twice MOP), with a nominal pass/fail criteria of 23 ml/min (0.00138 m³/hr). This is adapted from IGE/UP/1, but using hydrogen provides additional conservatism. The leak rates discussed in this study are very small in the context of ATEX and the gas industry.

Key results

- None of the anaerobic sealants showed any evidence of deterioration or leakage
- When used with PTFE tape, PTFE cord (string) or hemp fibres for support, none of the non-hardening compounds developed any leakage
- PTFE tape and PTFE cord used alone (without compound) did not seal well from the outset, but did not deteriorate over time
- Half of the non-hardening compounds used without support developed a leak rate above the failure rate in at least one test joint, though the leak rates were small
- Gas-rated commodity ball valves with NBR seals regularly developed leaks
- In all cases it is unclear whether hydrogen compatibility was the cause for failures

All five anaerobic jointing compounds tested did not develop leakage. These are:

- Cimberio C70-10
- Loctite 577
- Loxseal FR58-11 with Activator 11
- Loxseal FR58-11 alone
- Rocol Rapidseal

Four of the non-hardening jointing compounds tested without support material did not develop any leakage in any of the test pieces. Two more developed leakage at around the failure rate in one test piece, but the indications were that this was not caused by chemical reactions with hydrogen. The six non-hardening jointing compounds used on their own that did not show deterioration in contact with hydrogen were:

- Boss White
- Facot Silicon tape
- Fernox Hawk White
- Fernox Water Hawk
- Rocol Gasseal
- Stag Jointing Compound – Type B

The use of support material with non-hardening compounds appears to improve the reliability of threaded joints. None of the non-hardening compounds developed any leakage when used with a support material – including two that did develop leaks when used alone. There is not enough evidence to suggest that the cause of failure when support was not used was incompatibility with hydrogen.

Recommendations for further investigation

The cause for leakage developing over time in the non-hardening compound-only joints is not demonstrated to be hydrogen specific. Further investigation is recommended to determine whether methane or air would show the same effect in these sealants.

Two challenges encountered that highlight areas requiring further investigation are the ingress of air to installations with small leaks, and the suitability of commodity gas valves.

Contents

Document control

Executive summary

Contents

1	Introduction.....	1
2	Report structure	3
3	Sealant selection.....	4
3.1	<i>Sealant selection</i>	4
3.2	<i>Sealant list.....</i>	4
4	Test pieces.....	7
4.1	<i>Design of test joints - strings.....</i>	7
4.2	<i>Threads.....</i>	7
4.3	<i>Preparing the joints.....</i>	8
4.4	<i>String naming / reference</i>	8
4.5	<i>Joint creation.....</i>	8
4.6	<i>Baseline leak rates</i>	10
4.7	<i>Retiring joints</i>	11
5	Accelerated ageing.....	12
5.1	<i>Chemical compatibility and Arrhenius artificial ageing</i>	12
5.2	<i>Schedule of testing</i>	12
5.3	<i>Temperature control</i>	14
6	Test methods and experimental setups	17
6.1	<i>MPLR and failure leak rates</i>	19
6.2	<i>Leak testing</i>	20
6.3	<i>Leak test procedure.....</i>	22
7	Additional challenges encountered.....	23
7.1	<i>Week 1 test leaks</i>	23
7.2	<i>Locating leaks.....</i>	24
7.3	<i>Hydrogen retention / air ingress.....</i>	28
7.4	<i>Valve threaded connection leaks</i>	35
7.5	<i>Valve stem leaks.....</i>	37
7.6	<i>Method of retiring failed joints</i>	38
7.7	<i>Summary of test steps.....</i>	42
8	Results.....	44
8.1	<i>Experimental limitations - Arrhenius application.....</i>	45
8.2	<i>Overall results.....</i>	50
8.3	<i>Anaerobic jointing compounds (BS EN 751-1).....</i>	51
8.4	<i>Non-hardening jointing compounds with support material (BS EN 751-2).....</i>	53
8.5	<i>Non-hardening jointing compounds without support material (BS EN 751-2)</i>	54
8.6	<i>Unsintered PTFE tapes and cords (BS EN 751-3).....</i>	64
8.7	<i>Summary of results.....</i>	69
9	Conclusions and recommendations	72
9.1	<i>Sealant suitability.....</i>	72
9.2	<i>Use of support material with non-hardening compounds</i>	73
9.3	<i>Suitability of gas valves.....</i>	73

9.4 *Air ingress*..... 73

9.5 *Cause of leakage development* 74

10 Bibliography 75

11 Appendix A – Test Cards 76

1 Introduction

Sealant degradation has been identified as an evidence gap pertinent to conversion of existing networks from Natural Gas to hydrogen. This project aimed out to identify key prior art, provide an indication of how the guidance or regulations were developed, and provide evidence for whether commonly used sealants are likely to deteriorate and leak when used with hydrogen.

In Lot 1 of work package 7 in the Hy4Heat programme, Steer Energy measured relative gas leak rates from known failures such as incorrectly installed fittings, and joints assembled with no solder or sealant. Although threaded joints and PTFE tape were included, the study did not include differences in leaks caused by degradation.

Lot 2 of the Hydrogen Skills and Standards for Heat programme included testing of a large number of materials and components. It is unclear if sealants were included in this work.

A number of reports from the HSE work on the H21 project were shared at the kick-off of this project, and during the development of the proposal for this study. This work included a desk-based study of sealant compatibility, based on existing data. In many cases, compatibility was not known, so these sealants were deemed unsuitable, or unproven at this time. These documents include lists of sealants identified as being used on the gas network and in installations. This information was used to help develop the list of sealants to test to close this evidence gap.

Development of a rating system for potential effects of hydrogen on materials in 0-7 bar gas distribution assets (Bannister A. , 2022) is a key document within this suite, which provides information regarding testing development, and highlights issues with sealant testing with hydrogen. Specifically... *“A number of sealants defined by their trade names: the concern over these is the specific families of gases to which they have been tested and the applicable partial pressure of hydrogen. In general, the lack of detailed information for such products results in them being classified as ‘high’ potential degradation”*.

In that document, sealants are listed separately from jointing compounds. Initially in the section on ‘sealants & adhesives’, three categories are provided, which correspond to the three parts of BS EN 751 (anaerobic jointing compounds, non-hardening jointing compounds, and PTFE tapes). These appear to be generic for jointing compounds, with specific products then listed out separately in the sealants section. PTFE tapes are split into standard PTFE tape, graphite filled PTFE, and ceramic PTFE. These designations are not listed in BS EN 751-3. (BSI, 1997) (BSI, 1997) (BSI, 2022)

For anaerobic jointing compounds, the comment provided is... *“EN 751-1 Suitable for BS EN 437 G110 (50% H₂) and G112 (59% H₂) gases up to 5 bar: Other applications are outside scope of std. Not tested in 100% H₂. Equivalent hydrogen partial pressure for 59% H₂ at 5 bar is 3 bar.”* This is based on suitability, coverage in standards and product form/s, as found in literature by the authors.

A similar comment provided for non-hardening jointing compounds is... *“Class A compounds suitable for gas installations and BS EN 437 G110 (50% H₂) and G112 (59% H₂) gases up to 5 bar: Other applications are outside scope of std. Not tested in 100% H₂. Equivalent hydrogen partial pressure for 59% H₂ at 5 bar is 3 bar.”*

GIS (Gas Industry Standard) documents are cited for both types, but these are out of scope for this project.

A degradation rating is given for each type of material and product listed. This score is based on an assessment of the chemical compatibility and permeation, and likelihood of disbonding. Chemical compatibility and permeation refers to *“...whether the material is chemically compatible and does not allow significant quantities of gas to permeate through...”*, and disbonding is *“...the possibility that the interface between the sealant and the substrate (e.g. cast iron) could be degraded by hydrogen during its service life.”* The rating system is 0 - 25, with a higher score indicating greater risk.

The degradation rating for both anaerobic and non-hardening jointing compounds is 15.0, designated orange. This indicates medium degradation risk when in contact with hydrogen.

Standard PTFE tapes are noted as... “suitable to ISO TR 15916; Acceptable but high permeation as per ISO 1114-2”. Note that this is believed to be an error and should refer to ISO 11114-2. ISO 11114-2:2021 refers to gas cylinders and is therefore outside the scope of this study. Graphite filled and ceramic PTFE are also listed as suitable to ISO TR 15916. The permeation noted is expected to be at the very high pressures (>150 bar) associated with gas cylinders. All PTFE tapes are given a rating of 12.0, in the green category, indicating negligible degradation risk in contact with hydrogen.

Two of the products listed under sealants are given ratings of 14.0 and 15.5. This is in the orange category, indicating medium degradation risk. The other sealants are all in the red category with scores of 21.1 and 22.5, indicating high degradation risk. The comments indicate that none of these sealants have been tested in 100% hydrogen, so the degradation risk rating is believed to be based on the lack of information rather than positive indication of degradation.

Method for assessing material compatibility and component functionality for 0-7 bar gas distribution assets in hydrogen service (Bannister & Garrison, 2022) is believed to be based on information from the same literature search, and lists only the higher rated sealants.

In this document the degradation risk rating is scaled for the overall asset assessment method, with non-specific jointing compounds reduced from 15.0 to 12.5, and PTFE tapes reduced from 12.0 to 8.3. The risk categories do not change for any of the sealants or tapes listed; this change is only to provide mathematical consistency.

Overall, it is clear that there is a lack of information regarding sealant compatibility with hydrogen, although the comments indicate that some have been tested with partial hydrogen.

During development of the list of sealants to test in this project, some of the sealant manufacturers noted that some products were approved for use with hydrogen. A review of the techniques used was not carried out during this study, but this highlighted the difference between comparative leak testing and testing for chemical compatibility. This test programme is concerned with chemical compatibility.

2 Report structure

This report is arranged to provide a narrative and guide the reader through the development of the test method and adaptations to meet challenges.

Section 3 outlines the reasoning and justification behind which sealants were selected for testing by a working group of industry experts.

Section 4 provides information regarding the design of the test pieces (referred to as 'strings'). It goes on to detail how the joints were prepared, and provides the initial baseline leak rates.

Section 5 discusses the methodology adopted for compatibility testing, and why it was required. The test schedule and calculations of expected accelerated ageing is provided here.

In section 6 the leak testing method and setup is discussed. Justification for the leak rate pass threshold is given, with context of how the leak rates assessed compare to leaks of concern for ATEX exclusion zoning. The basic leak tests are described, and a basic procedure listed. Some of these steps are related to the concepts in section 7.

Section 7 presents a series of challenges encountered that shaped how the test programme developed, and what adaptations had to be made. In general the results are not shown here, but are used on occasion to define a point or provide evidence of successful mitigation. The responses and solutions to these challenges dictated how tests had to be carried out, which is then pertinent when interpreting the results.

The results are presented in section 8. Initially the overall results are given, and an assessment of the validity of the methodology. The main results are divided into groups to help with comparison between different types of sealant and sealant combinations. For the majority of strings there was no leakage, so only the overall increase in leak rate is provided. For strings where leaks did develop, graphs showing the trend of that leakage developing over time are shown, and a brief analysis carried out. The results also include a chart depicting the concentration of hydrogen recorded as remaining in the string immediately prior to leak testing. A test card for each sealant, showing the development of any leakage, is provided in Appendix A.

Section 9 contains the conclusions, summaries and some recommendations for where further investigation is required. These are linked to the challenges identified.

The sealants that did not appear to show deterioration due to hydrogen are listed in the conclusions. For sealants where this was not clear, the names are not provided.

3 Sealant selection

3.1 Sealant selection

The scope allowed up to 25 joint types to be constructed. It was important to capture the views of industry experts, to ensure that the sealants tested were appropriate, and representative of the existing pipework in UK installations. The aim during the sealant selection process was to ensure all stakeholders were satisfied that the samples tested would provide sufficient evidence for the complete range of options available in the open market.

A combination of products pertaining to parts 1, 2 and 3 of BS EN 751 were required. The majority of sealants used by gas fitters in the UK are believed to be of the non-hardening compound type, so these made up the majority of the test products.

A preliminary exploration into sealants produced a draft list that was suggested in the project proposal. This included reference to *Development of a rating system for potential effects of hydrogen on materials in 0-7 bar gas distribution assets* (Bannister A. , 2022) and common plumbing high street and online shops used by gas fitters. This was reviewed by industry experts, and additional sealants were incorporated at request. The final list included sealants suggested in GDN work instructions and was approved by HSE.

An attempt was made to contact the manufacturer for each chosen sealant, to ascertain whether hydrogen compatibility testing had been carried out, and obtain any additional information such as preferred installation methods and test procedures. Some of the manufacturers agreed to collaborate, and provided additional information regarding their sealants.

A two digit code was randomly assigned to each sealant or combination, to allow redaction of the results and minimise any bias. Preceding these digits was a 1, 2 or 3 to denote whether the string was within group 1 of anaerobic sealants (BS EN 751-1), group 2 of non-hardening compounds (BS EN 751-2), or group 3 of Unsintered PTFE Tapes (BS EN 751-3). This gave a three digit code for each sealant or combination.

The list of test sealants has been redacted from this report, with only the successful or recommended sealants named.

3.2 Sealant list

Throughout this report, particularly in the results section, there is reference to 'support' for the compounds. This refers to PTFE cord, PTFE tape, or hemp. This is generally a fibrous substrate that is more solid than the compound. The support forms a strong matrix that increases the pressure capabilities of a particular seal. The compound that is being supported reduces small leakage in the support matrix.

PTFE string and PTFE cord are the same, and BS EN 751-3 makes specific reference to this. The term 'cord' is used throughout this report to avoid confusion with the test strings.

Five anaerobic sealing compounds were tested that complied with BS EN 751-1. One of these was trialled with and without an activator for expedited cure time.

Eight non-hardening compounds complying with BS EN 751-2 were included without any support material. A silicone-based tape was also included in this group, although it was not a compound. A tenth sealant was included in this group, however it became apparent later that it was not manufactured to BS EN 751-2.

Three of the non-hardening compounds were also tested with PTFE cord as support, and two of the PTFE cords were included without compound.

White and yellow PTFE tape were included, both with and without compound. PTFE tape should not be used in conjunction with jointing compound, but it is expected that many gas engineers do this. White PTFE tape is not recommended for gas, but it is believed that this is still used currently, and has been used more commonly in the past. There is a range of thicknesses for different purposes, denoted by different colours.

Tape Jungle provides a breakdown of a wide range of available PTFE tape types as follows¹:

- White: Standard colour for tape used in basic plumbing jobs
- Yellow: Thread seal tape for gas lines such as natural gas, butane, and propane
- Pink: A more heavy-duty tape used for water lines
- Green: Grease-free tape used for oxygen lines
- Gray: Used for stainless steel pipes; thick tape to prevent seizing
- Red: Thick tape designed for pipes 1/2" to 2" in diameter and larger joints

RS Components notes that: *"Gas-rated PTFE thread seal tape is widely available, but it tends to be thicker and denser than standard PTFE tape for use in liquid plumbing environments. The latter can be prone to degradation and leaking if used inappropriately for gas pipelines, as it tends to be more porous. This is usually because of having been stretched thinner during the manufacturing process than the more heavy-duty PTFE tapes for natural gas ducting joints."*²

Finally, hemp and compound was added to the sealant list after the test programme had started. BS 6891:2015 precludes the use of hemp on low pressure domestic gas pipework, apart from in the specific case of existing long screw back-nut seals when used in conjunction with jointing compound. This was included to provide assurance regarding any existing examples. One of the non-hardening compounds that was used alone was also used with hemp. Preparation of a hemp and compound joint is shown in Figure 1.



Figure 1: Hemp and compound on a 1 1/2" black steel pipe thread

The complete list consisted of 25 sealants or combinations. While the brands and product names are removed, the full list of combinations is provided in Figure 2.

¹ <https://www.tapejungle.com/news/plumbers-tape-101-everything-you-need-to-know/#:~:text=Pink%3A%20A%20more%20heavy%2Dduty,in%20diameter%20and%20larger%20joints>

² <https://uk.rs-online.com/web/content/discovery/ideas-and-advice/ptfe-tapes-guide>

Line count	Anonymised reference code	Sealant or combination
1	101	Anaerobic jointing compound
2	102	Anaerobic jointing compound
3	103	Anaerobic jointing compound
4	104	Anaerobic jointing compound
5	105	Anaerobic jointing compound
6	206	Non-hardening jointing compound (207) with PTFE cord (205)
7	211	Non-hardening jointing compound (212) with PTFE cord (209)
8	213	Non-hardening jointing compound (201) with PTFE cord (not used alone therefore not listed)
9	216	Non-hardening jointing compound (207) with hemp
10	201	Non-hardening jointing compound without support material
11	202	Non-hardening jointing compound without support material
12	203	Non-hardening jointing compound without support material
13	204	Non-hardening jointing compound without support material
14	207	Non-hardening jointing compound without support material
15	208	Non-hardening jointing compound without support material
16	210	Non-hardening jointing compound without support material
17	212	Non-hardening jointing compound without support material
18	214	Non-hardening jointing compound without support material
19	215	Non-hardening jointing compound without support material
20	205	PTFE cord
21	209	PTFE cord
22	301	Thick PTFE tape (yellow)
23	302	Thin PTFE tape (white) & Non-hardening jointing compound (215)
24	303	Thin PTFE tape (white)
25	304	Thick PTFE tape (yellow) & Non-hardening jointing compound (215)

Figure 2: Sealant type and combination list

4 Test pieces

This section provides an introduction to the test pieces that were created for this study.

4.1 Design of test joints - strings

Three sets of four joints of each product or combination were assembled together in 'strings', with the test joints labelled A - D. This means there were 12 joints of each product in total. An exploded picture of an example string (with the updated valves and bonded washers) is shown in Figure 3.



Figure 3: String of 4 test joints

The original valves used had a 15 mm compression connection to the test equipment. This enabled a quick connection with relatively consistent seal. The valve end connected to the string was a ½" male parallel thread. This was not ideal as it should be tapered, but valves with this connection combination were not readily available.

The leak testing measured the leakage for the entire string, including the two additional joints for the valve connections. This was a compromise to meet the scope requirements.

Using female barrels with parallel threads ensured there was sufficient thread depth for the tapered male threads to engage with fully. Using reducing bushes allowed the use of 1" threads for the test joints but downsized valve connections. The same design was used for the hemp and compound strings, but with larger threads.

The joint labelling was consistent across all the strings. Each string has Valve 1 next to joint A, with joint D next to valve 2. The gas input was through valve 1 for the majority of the tests, although if the stem of Valve 1 was leaking, the string was sometimes rotated 180° to input through Valve 2.

4.2 Threads

BS EN 751 suggests brass threaded fittings for Natural Gas compatibility testing, so these were chosen for these tests. Fittings were procured from standard commercial plumbing stores, and conform to BS 10226-1. One-inch joints were used, with tapered male threads and parallel female threads. This size represents a compromise between thread gap size, and the quantity of fittings that could be tested.

The exception was the threaded joints for the hemp and compound. For these, malleable iron fittings with carbon steel pipes were used. These have rough threads, and are more likely to have been used with hemp and compound or putty in the past.

An example of one of the female malleable iron reducer threads is shown in Figure 4.



Figure 4: Rough threads on a malleable iron female reducer

4.3 Preparing the joints

All threads were cleaned using a wire brush and isopropyl alcohol (IPA) prior to application of sealants. This was done to remove any traces of cutting fluid, rust, paint, grease or other contaminants such as swarf or metal filings. This step is included on the majority of manufacturer installation instructions. The IPA was left to dry fully before assembly began.

4.4 String naming / reference

As noted in section 3.1, a 3-digit code was generated for each sealant or combination. A further preceding digit was added, to denote the temperature of storage. 20°C was a 2, 60°C was a 6, and 80°C was an 8. This produced a unique reference for each string, which could be cross-referenced but did not immediately identify the sealant.

The label was on the inlet (upstream) side for each string, allowing easy reference to each joint, and ensuring consistency with testing.

4.5 Joint creation

Following manufacturer instructions and notes in BS EN 751, sealant products were only applied to the male threaded part of joints.

All 150 valve to reducer units were assembled together. The same combination of PTFE cord and compound was used for these. Half of these units were then labelled.

The strings were assembled starting with the labelled valve-reducer unit, creating joint A by inserting a male nipple. Adding a female barrel connection to this was joint B; another male nipple the other side of the barrel became joint C; and the connection to the second reducer was joint D.

The original proposal suggested using two normal joints, and two 'sub-optimal'. The normal joints were A and B, with C and D the sub-optimal ones. The original plan for sub-optimal joints was to overfill the joint and only partially tighten it, however this led to the joints not being secure enough to remain consistent for long

term testing. The sub-optimal joints were therefore generally constructed by applying excessive sealant and allowing it to extrude. String 2208 during assembly is shown in Figure 5. The excessive sealant applied to the male thread of joint D is clear.



Figure 5: String 2208 during assembly, with excessive compound applied to joint D

Figure 6 shows string 6212 after assembly, before wiping away excess extruded compound. The difference between joints A and B, and C and D can be seen.

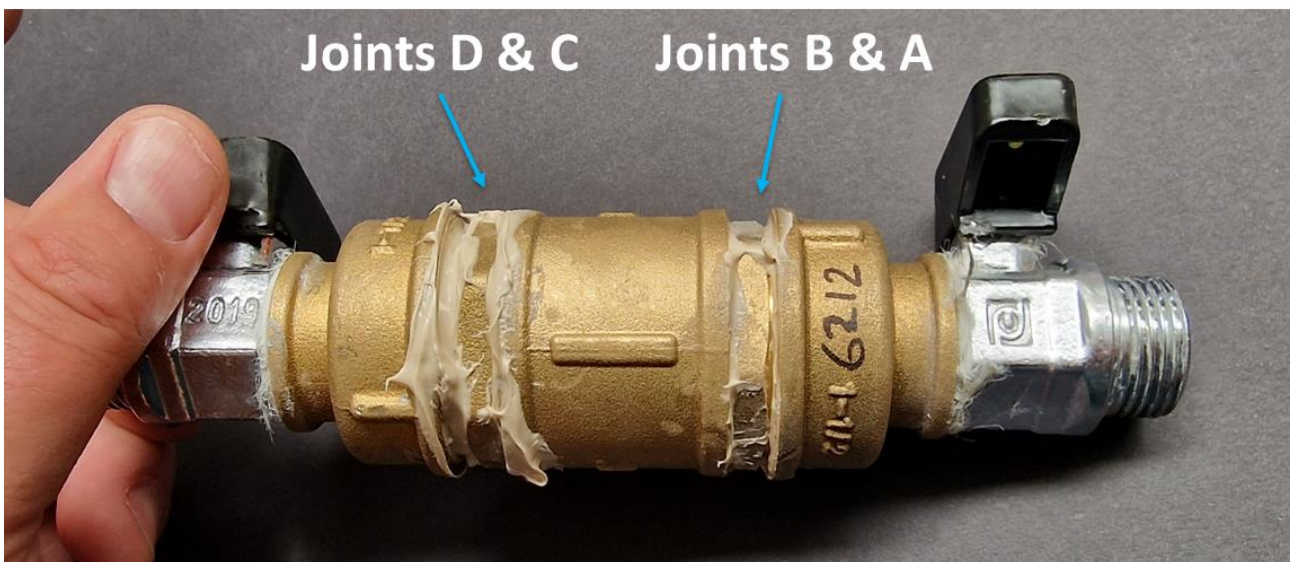


Figure 6: String 6212 after assembly

Additional information regarding how joints were constructed for testing under BS EN 751-2 was provided by one of the sealant manufacturers. The joint should be constructed using a layer of compound on the male threads, then PTFE cord, then a further layer of compound. This method was adopted for constructing the A and B joints that have both compound and cord, while the sub-optimal two joints in each of those strings were not given the first layer of compound.

Excess compound that was extruded around the joint was wiped away after assembly, though care was taken not to affect the test joints. After construction, the joints were left to cure for a minimum of 30 minutes at

ambient temperature, or longer if stipulated by the manufacturer instructions. The ambient temperature was in the region of 20°C.

4.6 Baseline leak rates

After assembly and curing or rest time, hydrogen leak tests at 150 mbar were carried out. These are referred to as the baseline leak rates, and are included in the main set of test results for each string.

Leak tests for the baselines were carried out in a similar way to the main test programme (see section 6 for more details and reasoning). The system leak rate was measured initially, for approximately 30 seconds. Valve 1 was then opened and the test piece charged from 130 mbar to 150 mbar. The total leak rate was then measured for 30 to 60 seconds after the flow stabilised. The system leak rate could then be subtracted from the total, to provide the test piece leak rate. An example is shown in Figure 7.

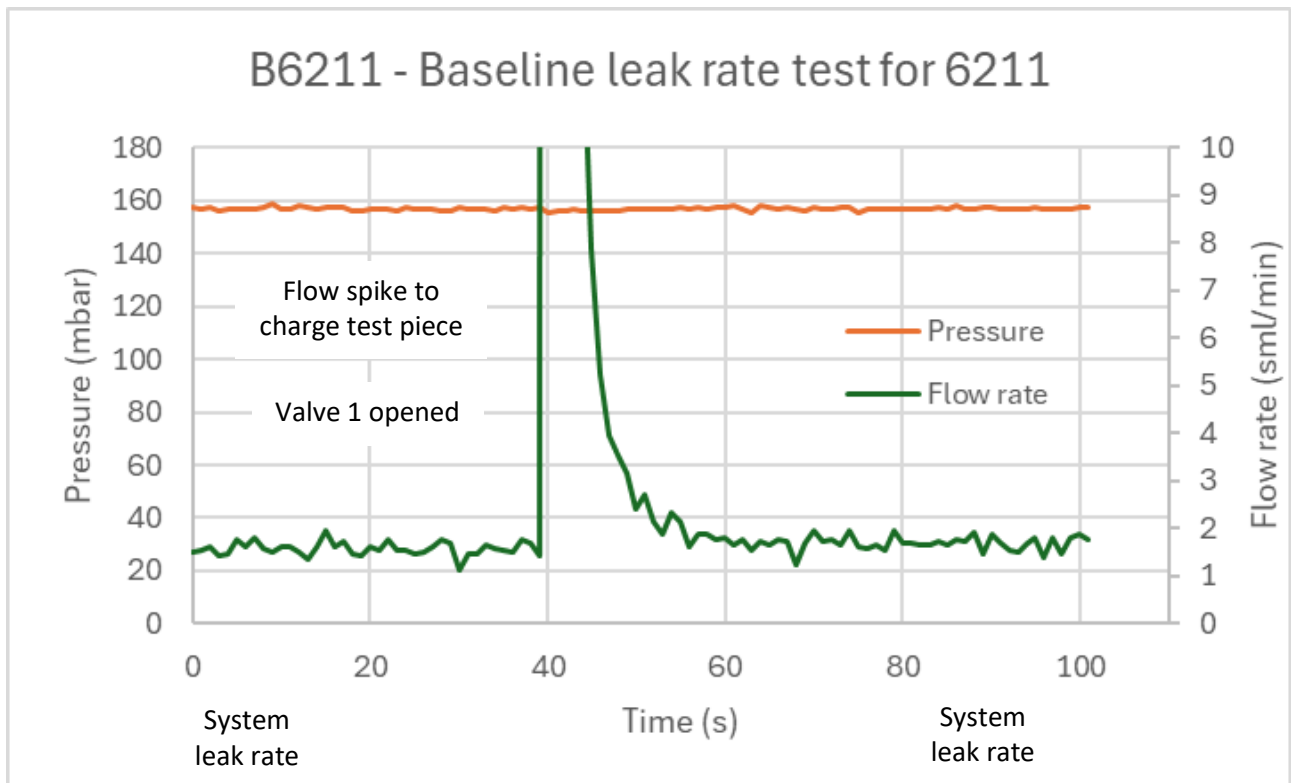


Figure 7: Baseline leak test for 6211

The strings that did show some leakage in the baseline tests are shown in Figure 8. Note that this differs from the leakage observed at week 1 tests, for which some strings were remade.

String reference	Date of baseline test	Leak rate at 150 mbar
8204	07/08/2023 15:02	2.0 ml/min (0.00012 m ³ /hr)
6205	03/08/2023 12:42	9.1 ml/min (0.00054 m ³ /hr)
6209	03/08/2023 11:12	6.7 ml/min (0.00040 m ³ /hr)
6301	04/08/2023 10:30	11.9 ml/min (0.00071 m ³ /hr)
8301	04/08/2023 10:39	13.2 ml/min (0.00079 m ³ /hr)
2303	04/08/2023 15:30	43.6 ml/min (0.0026 m ³ /hr)
6303	04/08/2023 15:40	348.6 ml/min (0.021 m ³ /hr)
8303	04/08/2023 15:52	288.7 ml/min (0.017 m ³ /hr)

Figure 8: Strings with baseline leakages

The majority of the strings did not leak at all. None of the hardening sealants under BS EN 751-3 showed any baseline leakage. 2105 leaked initially, but in retrospect this is most likely to be from a valve stem.

The strings highlighted blue in Figure 8 are from the group of non-hardening compounds without support material, while the orange highlighted rows are PTFE tape without compound.

It is notable that all three temperature strings for the thin PTFE tape (string 303) leaked from the start. Unsuccessful attempts were made to reduce these by tightening and remaking the joints. They were included in the test programme even though they were already above the failure leak rate, to identify whether the leakage increased over time.

4.7 Retiring joints

The overall aim of this study was to determine whether any sealants developed issues from contact with hydrogen. The initial plan therefore only included an assessment of pass or fail, and did not include investigating how high leak flow rates would reach.

The initially proposed plan was to remove any joints from the test programme if the leak rate increased above 23 ml/min. Once the joint was considered 'failed', the test could then be continued on the remaining joints in the string.

The process of removing a joint from a string is referred to as 'retirement' throughout this report. This initially involved applying external sealants, and progressed to fully disassembling the failed joints and remaking them so they were sealed and did not contribute to the measured leak rate for the string.

Further details on the retirement process, how it was developed, and the reasoning behind it are provided in section 7.6.

5 Accelerated ageing

5.1 Chemical compatibility and Arrhenius artificial ageing

The testing requirements in sections 2 and 3 of BS EN 751 require exposure to gas condensates for sealant compounds for use with Natural Gas. For this multiple tests are required, two assemblies stored for 72 hours at 20 °C with n-pentane and three assemblies with a 70-30 mixture of iso-octane and toluene. These induce faster chemical reactions of the same nature that would occur with Natural Gas, highlighting any incompatibility.

This is not applicable to hydrogen, as it is not a hydrocarbon. To replicate the chemical reactions caused by the gas condensates, the Arrhenius theory of reaction rate has been applied in this study. This is a proven method of artificially ageing materials, in which every 10°C above the baseline is equivalent to doubling the rate of chemical reactions. In this case this means degradation of the sealants over time, effectively doubling the representative age.

For these experiments, a baseline of 20°C was chosen to represent indoor ambient temperatures. 60 °C and 80 °C were chosen as the two other temperature to provide a distribution of aged samples.

80 °C was selected as the maximum temperature, as it was suitable for the majority of sealants while providing a reasonable project time-frame. This difference of 60 °C signifies a $2^6 = 64$ times increase in artificial ageing. At 80 °C, 50 years (2,600 weeks) can therefore be represented after 40.6 weeks. 60 °C is 40 °C above the baseline, which gives a scaling of $2^4 = 16$ times.

The time at which strings were removed from and returned to the ovens for testing was noted for each test. The temperature is approximated to 20°C for this period, and is included in the ageing calculation. This reduces the overall ageing, and the predicted age of 50.46 years was approximately 49.3 years once this was taken into account.

5.2 Schedule of testing

The schedule selected for testing in this study was designed to provide an even distribution of test samples, with an increase in data at the longest time. The temperature of the middle oven was increased to 60°C from the 50°C suggested in the proposal to provide more datapoints in the age range between approximately 6 and 12 years. The tests were scheduled to take place on Mondays and Thursdays, providing an additional working day to react to any challenges encountered.

Assembling the strings took more time than anticipated, leading to a second batch of strings being put into the ovens four days later. Some of the strings developed leaks within the first week. Instead of continuing with the retirement plan for those joints, those strings were disassembled and started again. These were therefore added to a third batch along with the additional hemp and compound strings, which were added to the scope late. Batch 3 was therefore slightly behind the other batches.

The updated and accurate schedule is shown in Figure 9, Figure 10 and Figure 11. The sample times provide a distribution of datapoints, with a maximum representative age of 50.46 years. This distribution provides a wide range of times, with a number of later datapoints. There are some deliberate duplications of test products at similar representative ages between different ovens, which provides a direct comparison between the temperatures.

The recorded test data included the time and date of testing, as well as the time and date each string was removed from and returned to its control temperature. This meant the exact timing of testing was not critical, and the schedule was adapted to meet changing requirements.

		Batch 1					Date in oven: 04/08/2023		
Actual Temperature (°C)	20°C			60°C			80°C		
	Week sampled	Date of sample	Age (weeks)	Week sampled	Date of sample	Age (years)	Week sampled	Date of sample	Age (years)
Strings in B1 2201, 2205, 2206, 2208, 2209, 2210, 2101, 2102, 2103, 2104, 2301, 6201 , 6202 , 6205, 6206, 6207, 6208, 6209, 6210, 6101, 6102, 6103, 6104, 6301, 8201, 8202, 8205, 8206, 8207, 8208, 8209, 8210, 8101, 8102, 8103, 8104, 8301	1	Thursday-10-Aug-23	1	1	Thursday-10-Aug-23	0.31	1	Thursday-10-Aug-23	1.23
	2	Thursday-17-Aug-23	2	2	Thursday-17-Aug-23	0.62	10	Thursday-12-Oct-23	12.31
	4	Thursday-31-Aug-23	4	8	Thursday-28-Sep-23	2.46	16	Thursday-23-Nov-23	19.69
	8	Thursday-28-Sep-23	8	16	Thursday-23-Nov-23	4.92	19	Thursday-14-Dec-23	23.38
	16	Thursday-23-Nov-23	16	19	Thursday-14-Dec-23	5.85	25	Thursday-25-Jan-24	30.77
	25	Thursday-25-Jan-24	25	25	Thursday-25-Jan-24	7.69	32	Monday-18-Mar-24	39.38
	32	Monday-18-Mar-24	32	32	Monday-18-Mar-24	9.85	36	Thursday-11-Apr-24	44.31
	40	Thursday-09-May-24	40	36	Thursday-11-Apr-24	11.08	40	Thursday-09-May-24	49.23
	41	Thursday-16-May-24	41	41	Thursday-16-May-24	12.62	41	Thursday-16-May-24	50.46

Figure 9: Batch 1 test schedule

		Batch 2					Date in oven: 07/08/2023		
Actual Temperature (°C)	20°C			60°C			80°C		
	Week sampled	Date of sample	Age (weeks)	Week sampled	Date of sample	Age (years)	Week sampled	Date of sample	Age (years)
Strings in B2 2202 , 2203, 2204, 2207, 2211, 2212, 2213, 2214, 2215, 2105, 2302, 2303, 2304, 6203 , 6204, 6211, 6212 , 6213, 6214, 6215, 6105, 6302, 6303, 6304, 8203, 8204, 8211, 8212, 8213, 8214, 8215, 8105, 8302, 8303, 8304	1	Monday-14-Aug-23	1	1	Monday-14-Aug-23	0.31	1	Monday-14-Aug-23	1.23
	2	Monday-21-Aug-23	2	2	Monday-21-Aug-23	0.62	10	Monday-16-Oct-23	12.31
	4	Monday-04-Sep-23	4	8	Monday-02-Oct-23	2.46	16	Monday-27-Nov-23	19.69
	8	Monday-02-Oct-23	8	16	Monday-27-Nov-23	4.92	19	Monday-18-Dec-23	23.38
	16	Monday-27-Nov-23	16	19	Monday-18-Dec-23	5.85	25	Monday-29-Jan-24	30.77
	25	Monday-29-Jan-24	25	25	Monday-29-Jan-24	7.69	32	Monday-18-Mar-24	39.38
	32	Monday-18-Mar-24	32	32	Monday-18-Mar-24	9.85	36	Monday-15-Apr-24	44.31
	40	Monday-13-May-24	40	36	Monday-15-Apr-24	11.08	40	Monday-13-May-24	49.23
	41	Monday-20-May-24	41	41	Monday-20-May-24	12.62	41	Monday-20-May-24	50.46

Figure 10: Batch 2 test schedule

		Batch 3					Date in oven: 21/08/2023		
Actual Temperature (°C)	20°C			60°C			80°C		
	Week sampled	Date of sample	Age (weeks)	Week sampled	Date of sample	Age (years)	Week sampled	Date of sample	Age (years)
Strings in B3 2202-3, 2216, 6201-1, 6202-1, 6203-1, 6212-1, 216, 8216	1	Friday 25-Aug-23	1	1	Friday 25-Aug-23	0.31	1	Friday 25-Aug-23	1.23
	2	Monday-04-Sep-23	2	2	Monday-04-Sep-23	0.62	9	Monday-23-Oct-23	11.08
	4	Monday-18-Sep-23	4	8	Monday-16-Oct-23	2.46	16	Monday-11-Dec-23	19.69
	8	Monday-16-Oct-23	8	16	Monday-11-Dec-23	4.92	20	Thursday-04-Jan-24	24.00
	16	Monday-11-Dec-23	16	20	Thursday-04-Jan-24	6.00	25	Monday-12-Feb-24	30.77
	25	Monday-12-Feb-24	25	25	Monday-12-Feb-24	7.69	31	Tuesday-26-Mar-24	38.15
	31	Tuesday-26-Mar-24	31	31	Tuesday-26-Mar-24	9.54	36	Monday-29-Apr-24	44.31
	40	Monday-27-May-24	40	36	Monday-29-Apr-24	11.08	40	Monday-27-May-24	49.23
41	Monday-03-Jun-24	41	41	Monday-03-Jun-24	12.62	41	Monday-03-Jun-24	50.46	

Figure 11: Batch 3 test schedule

The distribution of leak tests is given in Figure 12. The schedule was arranged to provide redundancy and duplication of some data points, while providing an even distribution of time and two data points close to the longest time.

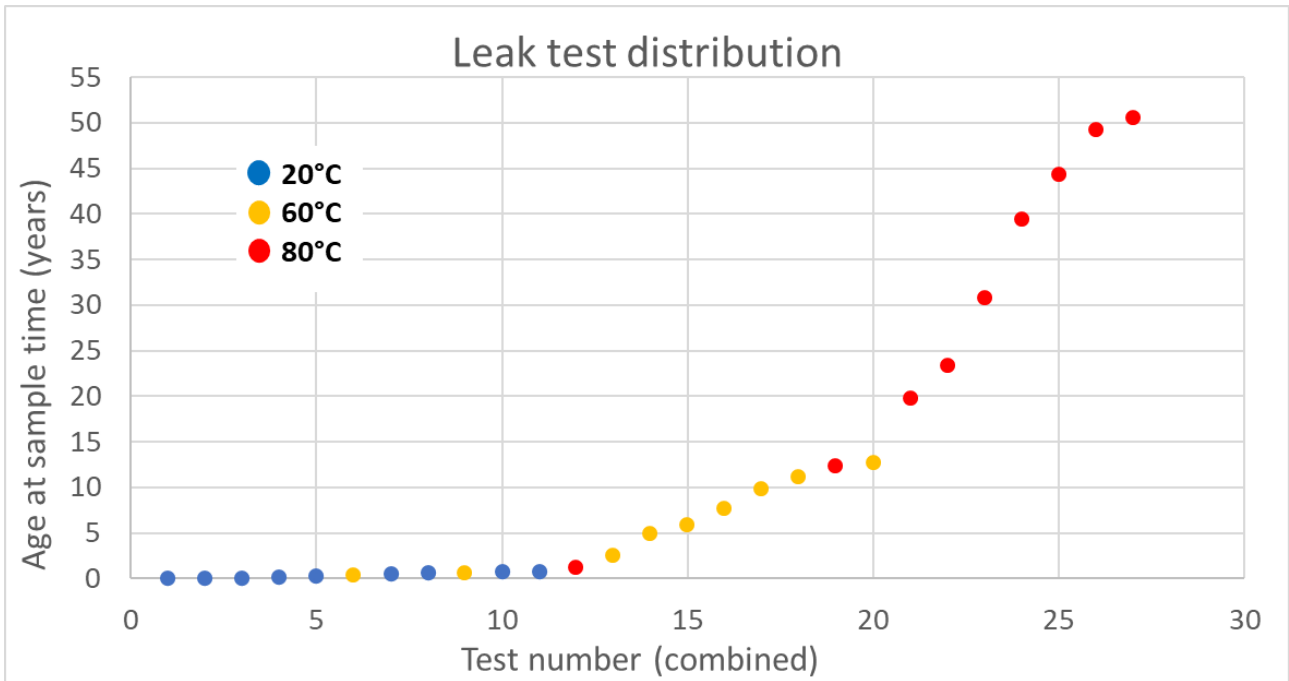


Figure 12: Test sample distribution

The 20°C strings are differentiated in in Figure 13, with the time axis scaled to weeks.

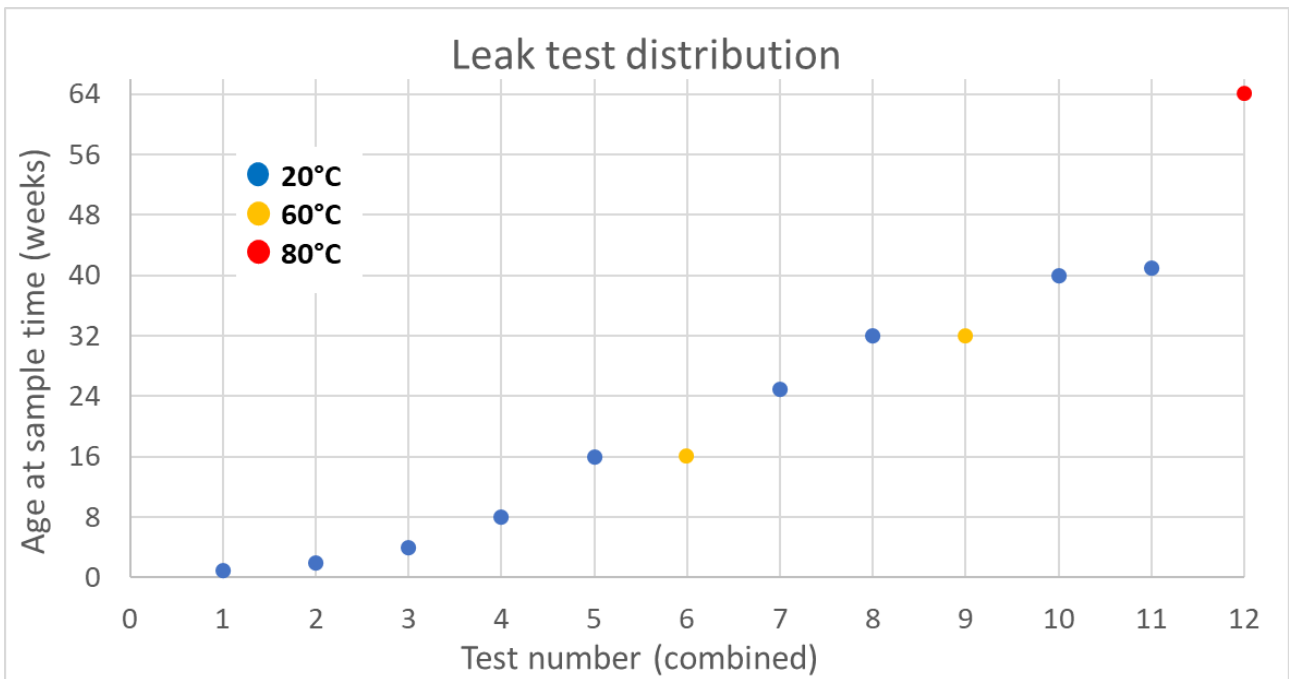


Figure 13: Test sample distribution - scaled to weeks

5.3 Temperature control

Two laboratory ovens were used to control the strings at 60°C and 80°C, while a laboratory incubator was used for the 20°C strings. This report refers to all three as ovens, though the incubator had to provide both heating and cooling.

The temperature in each of the ovens was logged at 15 minute intervals for the duration of the test programme. The logging devices were inside the ovens, with an external probe extension to download readings regularly.

The equipment used was:

- LMS Model 80 cooled incubator - set at 20 °C
- Binder BF 56 (E3.1) laboratory oven - set at 60 °C
- Binder FD 56 (E3.1) laboratory oven - set at 80 °C
- 2 x DS1922L Thermochron iButton - logging 15 min intervals (20 °C and 60 °C)
- DS1922T Thermochron iButton - logging 15 min intervals (80 °C)

The ovens and incubator are shown in Figure 14. Fire suppression and additional peripheral thermal cutouts were included.



Figure 14: Laboratory ovens and incubator for maintaining consistent temperatures

It should be noted that the joints were subjected to thermal cycling, by taking out of the ovens and cooling. BS EN 751 includes a thermal cycling component, so the sealants were expected to endure this.

5.3.1 Challenges encountered with temperature control

During the test programme the incubator occasionally lost control and the temperature fluctuated. This was sent for repair over the winter, while the 20°C strings were placed in a warmed temperature controlled enclosure. This was successful, and they were returned to the incubator in due course.

These temperature fluctuations can be seen in the trace for the strings, and are included in the calculations for the Arrhenius ageing. The fluctuations caused by incubator malfunction are annotated in Figure 15.

It should be noted that the iButton temperature loggers have a smaller thermal mass than the strings, so record fluctuations in temperature more quickly than the actual change in the strings. When the 20°C strings were moved to the heating only box, the iButton was placed next to a spare string and wrapped with insulation. This provides a more accurate representation of the temperature cycling seen by the strings, but space restrictions in the ovens generally precluded this.

This can also be seen in the temperature trace when the door is opened and the strings removed for testing. The temperature loggers remain in the oven during this period (but are discounted from the ageing calculation). Spikes and dips in temperature are seen at these points, but these do not represent true temperature fluctuations of the strings.

5.3.2 Temperature logs

The final temperature traces are shown in Figure 15, with annotations for the 20°C incubator to show the changes made due to fluctuating temperatures.

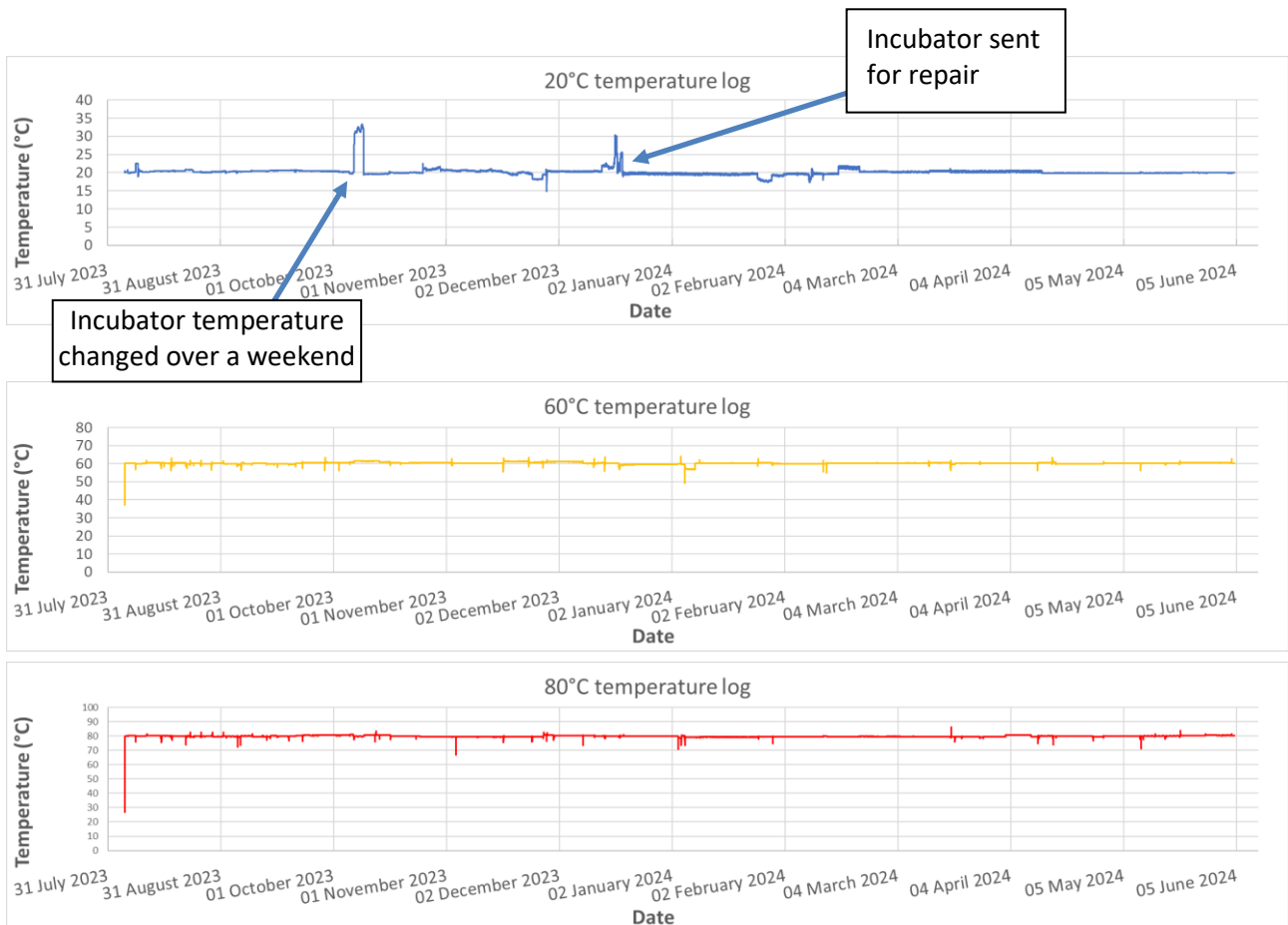


Figure 15: Temperature traces for 20°C, 60°C and 80°C ovens

6 Test methods and experimental setups

The primary aim of this study was to establish whether hydrogen affects sealants over time. To see this change in leakage over time was monitored. The scope for this work was downstream with a maximum operating pressure (MOP) of 75 mbar.

The testing process deviated from a simple leak test at scheduled intervals due to complications discovered during the test programme. A summary of the resulting decisions and procedures is provided in Figure 16. This is a simplified flow chart, based on the decisions made towards the end of the test programme.

The flow chart is split into two sections – initial string setup, baseline tests and week 1 tests, then the remaining test programme.

All planned activities are shown in blue, with delay periods corresponding to the Arrhenius model in green, additional steps taken in orange, and decisions in grey. Information flow is depicted by dotted lines, with actual string progression shown with solid lines.

The differentiation of action based on leak rate above and below 23 ml/min was not strict. Leakage above 10 ml/min was generally investigated, however as many of these steps were beyond the initial project scope, it was dependant on workload and available time. Leak rates above 23 ml/min were always investigated.

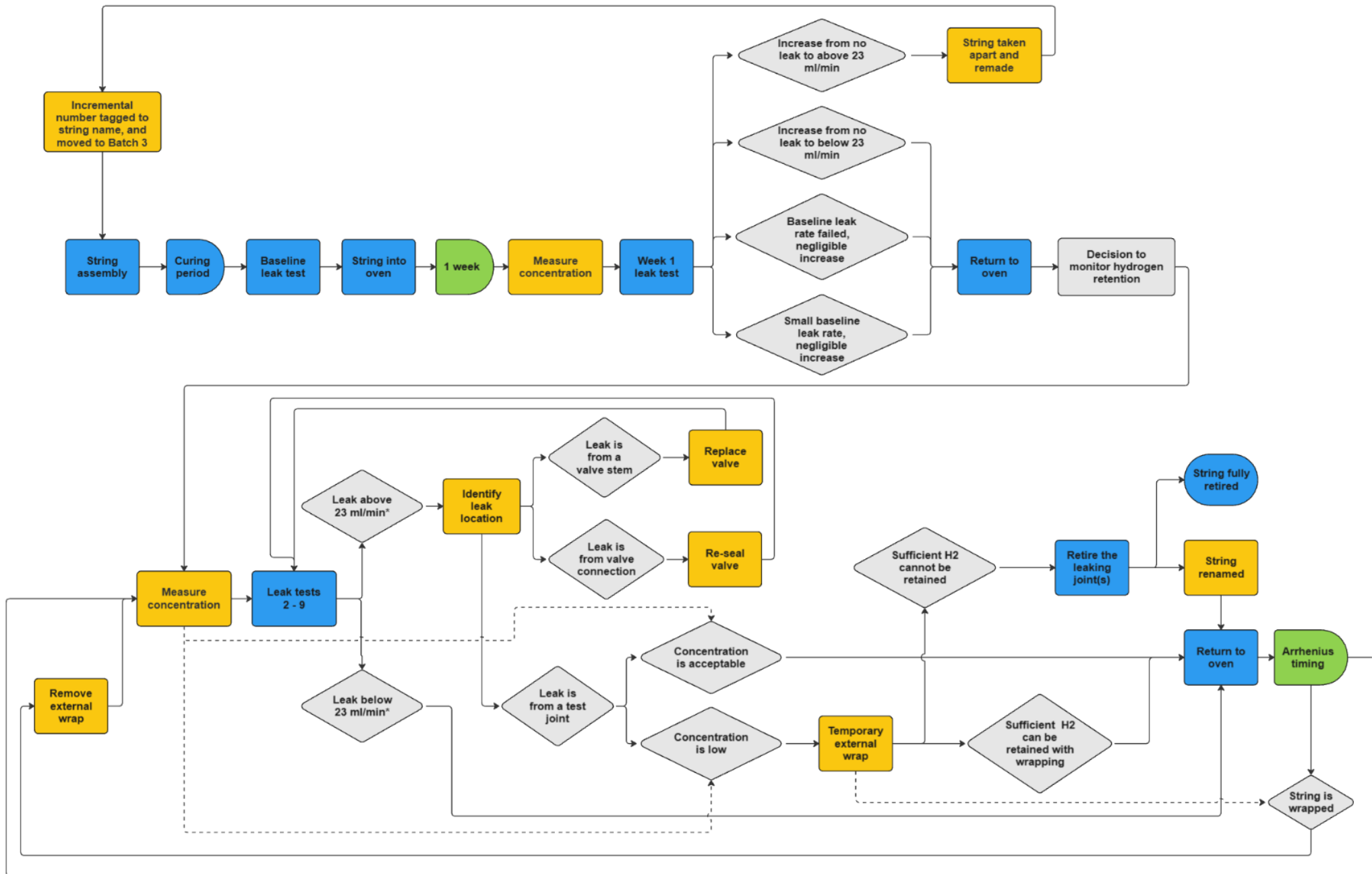


Figure 16: Flow chart of decision tree and processes

6.1 MPLR and failure leak rates

The maximum permitted leak rate (MPLR) is the proposed threshold above which a joint is considered to fail. Practically, the experiment for a particular string could not be continued if leak rates were too high, as hydrogen could not be guaranteed to be sufficiently present to ensure that compatibility was being tested. This is discussed further in section 7.3.

BS EN 751-1/2/3 does not stipulate measuring a leak rate following the gas condensates test, but instead the joint should be submerged in water and pressurised to 5 bar (BSI, 1997) (BSI, 1997) (BSI, 2022). After an initial settling period of one minute, any bubbles indicate leakage and the test is considered a failure. For this study, the MPLR for Natural Gas in IGE/UP/1 was used as a guide, as submerging in water had the potential to introduce side effects when considering long term compatibility (IGE/UP/1 Ed. 2. , 2005).

In IGE/UP/1, MPLR is based on energy release rate, with a maximum allowed of 0.054 MJ/hr at operating pressure. For new installations and extensions using Natural Gas, this corresponds to a leak rate of 0.00138 m³/hr (0.023 l/min). HSE research report RR1169 discusses MPLR in the context of leak hazard (Gant S., 2022). Table 3 in section 5 provides theoretical flow rates of methane and hydrogen that provide equivalent energy release rates. To provide an energy flow rate of 0.054 MJ/hr, methane must flow at 0.0014 m³/hr, while hydrogen flow rate must be 3.2 times as high, at 0.0045 m³/hr.

For a given pressure, hydrogen does leak at a higher volumetric leak rate than methane, but not as much as 3.2 times. For larger leaks with high Reynolds value and turbulent flow this is with a factor of up to 2.82 times, but for small leaks with low Reynolds value and laminar flow it is a factor of 1.24 times. (Ryan & Roberts, 2020) At 2.8 times the volumetric flow rate, hydrogen releases approximately 90% of the energy flow, and at 1.24 times approximately 30% of the energy is released.

In practical terms the Reynolds number and therefore the gas flow profile through a leak is dependent on a number of factors such as hole shape and length. A comparative leak test was carried out on string 2201, which had previously shown a hydrogen leak rate of 24.7 ml/min (0.0015 m³/hr). With methane, the leak rate was 11.8 ml/min (0.00071 m³/hr), and with air it was 8.4 ml/min (0.00050 m³/hr). When tested again with hydrogen, the leak rate had reduced to 19.5 ml/min (0.0012 m³/hr). Depending on the figure used, the volumetric leak in hydrogen was between 1.7 and 2.1 times that of methane, indicating the flow profile is therefore likely to be in transition between being laminar and turbulent.

Applying the same ratio of volumetric leak rate to the MPLR selected for hydrogen gives a flow rate for methane of between 0.000657 and 0.000812 m³/hr, and an energy flow between approximately 0.025 and 0.031 MJ/hr.

In terms of hazard assessment, RR1169 also explores the likelihood of a flammable atmosphere being generated by a leak in a small cupboard. This is determined by the volumetric flow rate, not the energy flow rate (with variables such as diffusivity also playing a part). RR1169 therefore makes the recommendation to equate the volumetric flow rate of the gases to determine the maximum allowed physical leak size. This is the same principle applied in this study, although as the leaks at the MPLR threshold are in transition between laminar and turbulent, additional conservatism is included for hydrogen in terms of energy released.

These MPLR selected can also be assessed in the context of DSEAR hazardous leaks. Based on Steer Energy's orifice flow calculations, a 0.25 mm² hole with discharge coefficient of 0.6 would provide a hydrogen flow rate of approximately 0.32 m³/hr at 150 mbar. A 0.025 mm² hole with discharge coefficient of 0.6 would provide a hydrogen flow rate of approximately 0.03 m³/hr at 150 mbar. While there are inaccuracies in the estimated flow rates – particularly due to the transitional nature of the flow regime and the unknown nature of the coefficient of discharge – the threshold in this study is an order of magnitude less than this. The largest leak observed was 0.024 m³/hr, which is approaching the leak rate expected from a 0.025 mm² hole. The MPLR selected of 0.0138 m³/hr is estimated to be the result of a hole in the region of 0.06 mm diameter, or 0.0028 mm².

The other aspect when considering the hazard of a leak is the dimensional limits of ignitability. Models referenced in RR1169 indicate the relative extent of a hydrogen plume (to LFL, from a low pressure source and

not choked flow) to be between 1.5 and 2.3 times that for methane. Experiments carried out by Steer Energy in Ignition Probability in Buildings Associated with Small Services (Roberts, 2023) demonstrated similar findings for actual ignitable limits, noting that the concentration at ignition differed.

All these results are based on equivalent leaks from the same hole. Effectively reducing the hole size for hydrogen to match the flow rate of methane will reduce the plume extent. It is expected that as the leak flow is within the transitional period from laminar to turbulent, reducing the hole size further would mean the hydrogen leak would become more laminar. This complicates the direct comparison, as the coefficient of discharge changes, affecting the flow profile and therefore potentially air entrainment and concentration decay.

In summary, using the same volumetric flow rate for hydrogen as IGE/UP/1 uses for Natural Gas ensures the physical hole size for an acceptable leak is smaller. This adds conservatism to the results. The leaks are small enough to not be of concern for the accumulation of explosive atmospheres, but may still be lit directly.

It should be noted that this study is concerned with deterioration of sealants, rather than the specific leak rate. While a failure rate has been applied, the specific rate is not critical to the findings in these experiments.

6.2 Leak testing

Leak rates can be inferred by monitoring pressure decay over time, or by measuring input flow at a regulated pressure. The former relies on stable temperatures and accurate measurement of the enclosed volume. These can introduce sensitivity to external factors, especially with a small enclosed volume. For example, placing a finger on one of the strings produces a measurable increase in pressure.

The leak rates are given in ml/min as this was the recording format and is appropriate for the level of leakage and precision used. The summary of results also presents these leak rates in m³/hr, to provide context within the gas industry. It should be noted that the leaks rates seen were very small in the context of ATEX and the gas industry.

Steer therefore measured the flow rate by providing a regulated supply pressure and monitoring the input flow. This method is reliant on the pressure regulator opening, which requires a minimum flow rate. A system leak of approximately 1 ml/min (0.00006 m³/hr) was therefore allowed, measured, and subtracted out in post processing.

The primary flow meter used was a *Bronkhorst F-201CV-200*. This had a maximum flow rate of 200 ml (0.012 m³/hr), and with a measurement turndown of 100:1, could accurately measure flows around 2 ml/min (0.00012 m³/hr). In practice, above approximately 0.3 to 0.4 ml/min is differentiable from noise, although the accuracy is decreased. A second meter with higher maximum flow was used for the leakier strings. With a 100:1 turndown from 10,000 ml/min (0.6 m³/hr), this provided useful readings from a minimum of approximately 100 ml/min (0.006 m³/hr). System leak rate (without test piece attached) was generally in the region of 1 ml/min, and this was read by the larger flow meter as approximately 30 to 40 ml/min. Combining the two results to provide an estimate of leak reduction during retirements was therefore challenging, and introduces more error to otherwise precise readings.

The leak testing equipment used was:

- 2 x Compa Novacommet BP1813 adjustable pressure regulator
- Bronkhorst F-201CV-200 flow meter – 2 to 200 ml/min (0.00012 to 0.012 m³/hr) in hydrogen
- Bronkhorst F-201CV-10K flow meter – 100 to 10,000 ml/min (0.006 to 0.6 m³/hr) in hydrogen
- IFM PG2457 pressure gauge – up to 1 bar
- SteerLogger2 – bespoke data logger set to 1 second intervals
- wHystle purge indicator

Two pressure regulators were used. One was used for the leak testing, and was set at approximately 150 mbar. A second regulator was used to purge and charge the system and test piece to approximately 130 mbar. The difference to the test pressure ensured a burst of flow when valve 1 was opened. This provided a

clear distinction between the system only leak test and the leak test of the total installation including the test piece.

A simple flow chart detailing the leak test setup is shown in Figure 17.

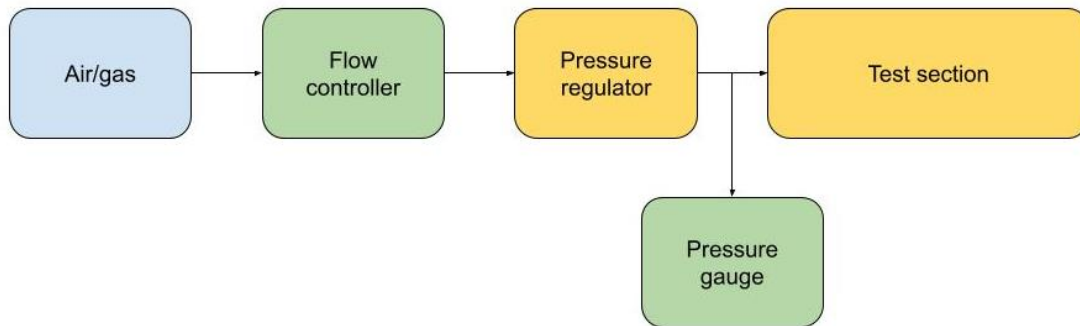


Figure 17: Leak testing setup

The pressure and flow rate for the leak tests were logged. Due to the substantial quantity of data produced however, the results presented are based on manual records of the outputs. The raw logged data is available for reference if required.

The dual hydrogen 130 and 150 mbar supplies with pressure gauge and test string inserted is shown in Figure 18.



Figure 18: Test setup with twin hydrogen supplies

The hydrogen supplies, control and logging equipment are shown in Figure 19. During testing, both flow controllers were set to 100%, and used only as meters. Individual flow was controlled using isolation valves.

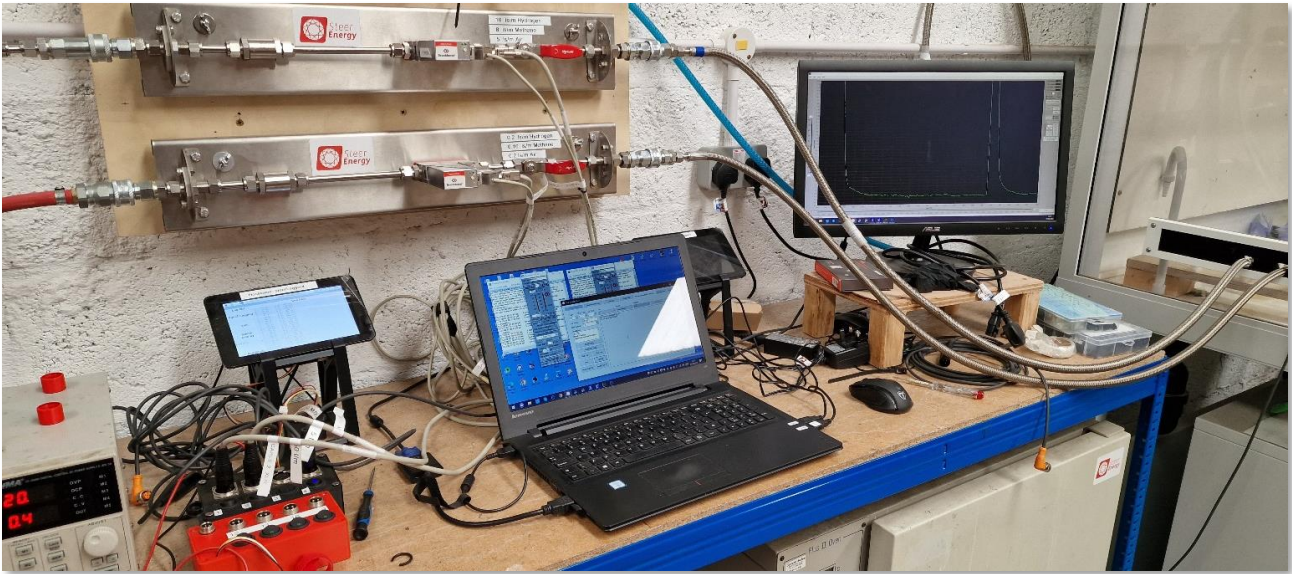


Figure 19: Hydrogen supply and logging equipment

6.3 Leak test procedure

This section outlines the key steps taken in the testing process.

Steps 3, 9 and 11 (highlighted by orange text) were unplanned, and added during the test programme as a response to challenges encountered. These developments are detailed in chapter 7.

The principal stages were:

1. Remove strings from ovens at allocated times
2. Allow to cool to ambient temperature ($20^{\circ}\text{C} \pm 5^{\circ}\text{C}$)
3. Measure concentration of hydrogen retained (logged separately)
4. Purge test pieces to hydrogen and confirm using purge whistle
5. Close valve 2, charge test piece to 130 mbar and isolate valve 1
6. Measure system leakage at 150 mbar for minimum 30 seconds
7. Open valve 1 and wait for leak rate to stabilise
8. Measure total leak rate at 150 mbar for minimum 30 seconds
9. If leakage is above or approaching the failure rate, attempt to locate where leakage is coming from
10. Close valve 1, leaving the string charged to 150 mbar
11. If leakage is above or approaching the failure rate, temporarily wrap leaking joints externally (remove again for leak testing)
12. Return string to the oven

7 Additional challenges encountered

As the test programme progressed, various challenges were encountered and experimental processes had to be adapted accordingly. The issues were:

- Leaks developing at week 1 tests
- Locating leaks
- Hydrogen retention (air ingress)
- Valve connection leakage
- Valve stem leakage
- Leaking joint retirement

These issues are discussed briefly in the following chapters, as the resolution dictated how the test programme developed. Some of the findings are important from a safety perspective, and highlight the need for additional investigation. These have been identified and are highlighted in section 9.

A summary of the decisions made and steps taken is provided at the end of this chapter.

7.1 Week 1 test leaks

Some of the strings began to leak by the time of the first round of leak tests (note this is approximately 1 week after the baseline leak tests). The results and proposed solutions were discussed with the steering group, and the actions agreed are shown in Figure 20.

String reference	Problem	Action taken
6201, 6202, 2202, 6203, 6212	Increased from no leak to failure leak (above 23 ml/min)	Strings remade, added to batch 3 with incremental numbering
2203, 6214, 8203, 8214	Increased from no leak to leak below 23 ml/min	Continue with scheduled testing, monitor hydrogen retention
2303, 6303, 8303	All joints failed baseline leak rate. Increase was negligible.	Continue with scheduled testing, monitor hydrogen retention
6205, 6209, 6301, 8301	Baseline moderate leakage (below 23 ml/min) - week 1 shows slight variation but no failures	Continue with scheduled testing, monitor hydrogen retention

Figure 20: Early test leaks

6201, 6202, 2202, 6203, 6212 were disassembled and reconstructed with an incremental number to differentiate from the original string. These strings were added as batch 3. New fittings were used for each, to eliminate the chance that poor threads were the cause of leakage.

There was generally no clear indication why the strings had developed leaks, but 6202 had an obvious leak through a visible hole in the compound. This is shown in Figure 21.



Figure 21: 6202 leak path after one week

The strings with thin PTFE tape (reference ending 303) was clearly inferior at sealing than the strings with sealants, compounds or combinations, and did not seal well from the start. The other plain PTFE tape was code ending 301, which showed moderate baseline leak rate in two of the strings.

202 sealant presented challenges to assemble strings without leakage. String 2202 was remade several times, and was ultimately labelled as 2202-3.

The final baseline leak rate for all the remade strings was 0.0 ml/min at 150 mbar.

7.2 Locating leaks

Measuring the leakage of a string includes four test joints, two valve connections, leakage through valve stems, and the isolation of valve 2. Identifying where any leakage came from was important for understanding whether the sealants were degrading.

The primary options generally used for locating leaks are:

- Leak detection fluid
- Submersion in water
- Use of a gas sniffer

A gas sniffer was used, as it was the least intrusive to the joints and exposed sealants. Although no adverse reaction would be expected, avoiding the additional variable of adding chemicals or moisture was beneficial.

Initially a *Testo 316-2* gas sniffer was used to identify where leakage was coming from, but this was not accurate. As the test programme progressed, the methods of locating where hydrogen was escaping from were upgraded. Eventually, the leaking joint could be identified with reasonable accuracy and reliability, and an estimate how much of a leak could be attributed to each joint.

During the final leak tests, leak detection fluid was used to identify leaking joints.

7.2.1 Submerging valve connections

Some of the leakage detected was clearly coming from the valves, particularly Valve 1. Before the locating method was fully refined, an attempt was made to demonstrate this by submerging the valve connection under water, and observing for escaping bubbles.

The standard method of leak detection outlined in BS EN 751 following gas condensates compatibility testing is to submerge the test joint in water, and observe for any bubbles (BSI, 1997) (BSI, 1997) (BSI, 2022). The test piece is submerged in water for 5 minutes, and after an initial stabilisation period of 15 seconds, any bubbles observed is considered a failure. It is not specified in BS EN 751, but it is expected that bubbles produced by leakage would detach from the joint surface and rise in the water.

An attempt to replicate this was made, to show leakage from valve connections on some of the strings. The setup for this experiment is shown in Figure 22. Care was taken to keep all other joints dry.



Figure 22: Submerged valve connection

A closer view of this experiment on string 2303 is shown in Figure 23. Two bubbles did appear, seeming to come from the joint that had been identified as leaking. These bubbles did not detach and float to the surface however.



Figure 23: Observing bubbles from submerged valve connection on string 2303

This was an unexpected result, and could indicate a limitation for bubble testing in water with hydrogen for small leaks. This was not explored further, but did necessitate an improvement in the leak identifying process for this study.

7.2.2 Identifying leaking joints

A way to more accurately identify where leakage was coming from was required. A *Testo 316-2* gas leak detector was used. This has high sensitivity to hydrogen, detecting down to 10 ppm.

The escaping hydrogen spreads rapidly, and detecting exactly where it is leaking from was not possible. Ultimately, a series of baffles were designed, manufactured and fitted to the string shape, minimising interference between different joints. The final version is shown in Figure 24, attached around a sample test string, with bespoke shapes to fit both sizes of circular barrel and the hexagonal nipple flats.



Figure 24: Baffles to aid locating leaks

The final setup for leak locating is shown in Figure 25. While the proximity of joints made precise readings difficult, the location where concentration was strongest could be identified with greater precision. Leaking joints could therefore be prioritised for temporary external sealing (see section 7.3.2), or permanent retirement (see section 7.6). Using this system made it possible to determine that some of the leakage coming from the valves was actually from the valve stem. This is discussed further in section 7.5.



Figure 25: Refined method for identifying leaking threads

7.3 Hydrogen retention / air ingress

Retaining a high concentration of hydrogen was important for the accuracy of the Arrhenius artificial ageing model, as it proves that the sealant was in contact with hydrogen.

The strings were left pressurised with hydrogen (nominally 150 mbar) after each leak test. As the strings contain a very small volume, no pressure was expected to be retained in the strings between leak tests. The project scope and budget did not allow for a constant positive pressure hydrogen feed to be applied to the strings. This was considered an unnecessary expense and hazard, as the hydrogen concentration was expected to remain at 100%.

An additional step was included for the first round of leak tests carried out at week 1, to confirm and provide evidence that the concentration of hydrogen retained in the string remained at 100%. The results of this showed that the hydrogen concentration was substantially lower for several of the strings. To maintain validity of the test programme, the strings were purged on a regular basis, and an exploration into the cause was undertaken. The concentration measurement step was adopted for the rest of the test programme, and was carried out immediately prior to the scheduled leak test for each string.

This section details how the concentration measurements were carried out, and discusses the results and implications of the findings for the test programme. Further discussion on the wider implications are provided in the overall discussion section.

7.3.1 Measuring hydrogen concentration in strings

To measuring the hydrogen retention a VQ456MR sensor was connected to a bespoke logger developed by Steer, and calibrated for either hydrogen or methane.

The hydrogen sensor was fitted to a ½" BSP barrel using vacuum putty and leak sealing tape. This could then be screwed onto the valve of a string, to provide an indication of the hydrogen concentration retained in the string prior to leak testing. This setup is shown in Figure 26.



Figure 26: VQ456MR hydrogen sensor attached to a test string

A series of calibrations were carried out to ascertain the readings expected for a range of concentrations. The sensors are calibrated individually by Steer and used for a variety of projects, however one was set aside to use for the duration of this study. The maximum readings are sensitive to a range of external influences, but the response curve remains relatively stable. The response was scaled for each set of tests to ensure readings are valid.

This is done by applying pure hydrogen directly into the sensor at the beginning of each batch of tests. This initial spike is used for the first normalisation, which changes over time. In Figure 27 note the 100% spike to the far left.

The original readings for the week 1 tests are shown in Figure 27. A single log was used for a full set of results, identifiable by time and sequence.

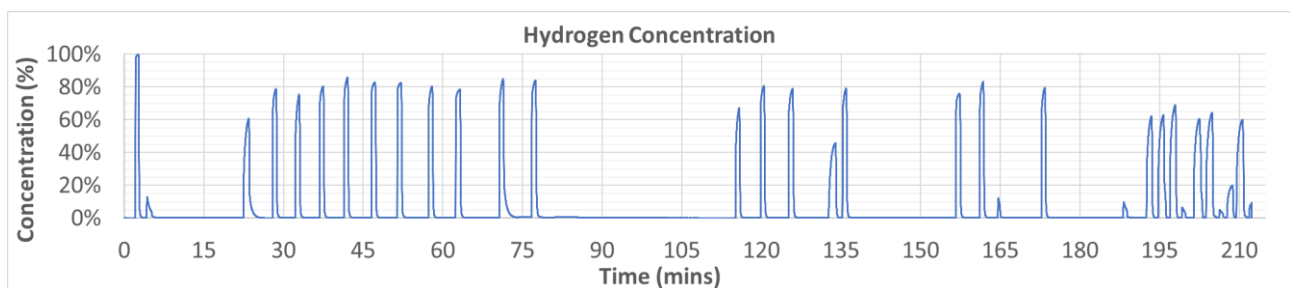


Figure 27: Week 1 concentration checks prior to secondary normalisation

The rest of the spikes peak at approximately 80% to 85%. Attaching the sensor to a string always leaves a cavity filled with air. This could not realistically be purged to the gas mixture that is in the string. This prompted the need to conduct a second calibration to account for the experiment limitations of fitting the sensor to a string. A spare string was purged to a range of known concentrations, and the sensor attached in the way it would be for a test. The peak output reading (in mV) was thus normalised to a string concentration.

While this first set of results was taken with the string (and therefore sensor) horizontal, the calibration and subsequent string tests were done in a vertical orientation, to make concentration readings faster and more consistent. The strings were stood on the end of valve 1, with the sensor attached to valve 2. The original hydrogen concentration test setup is shown in Figure 28.

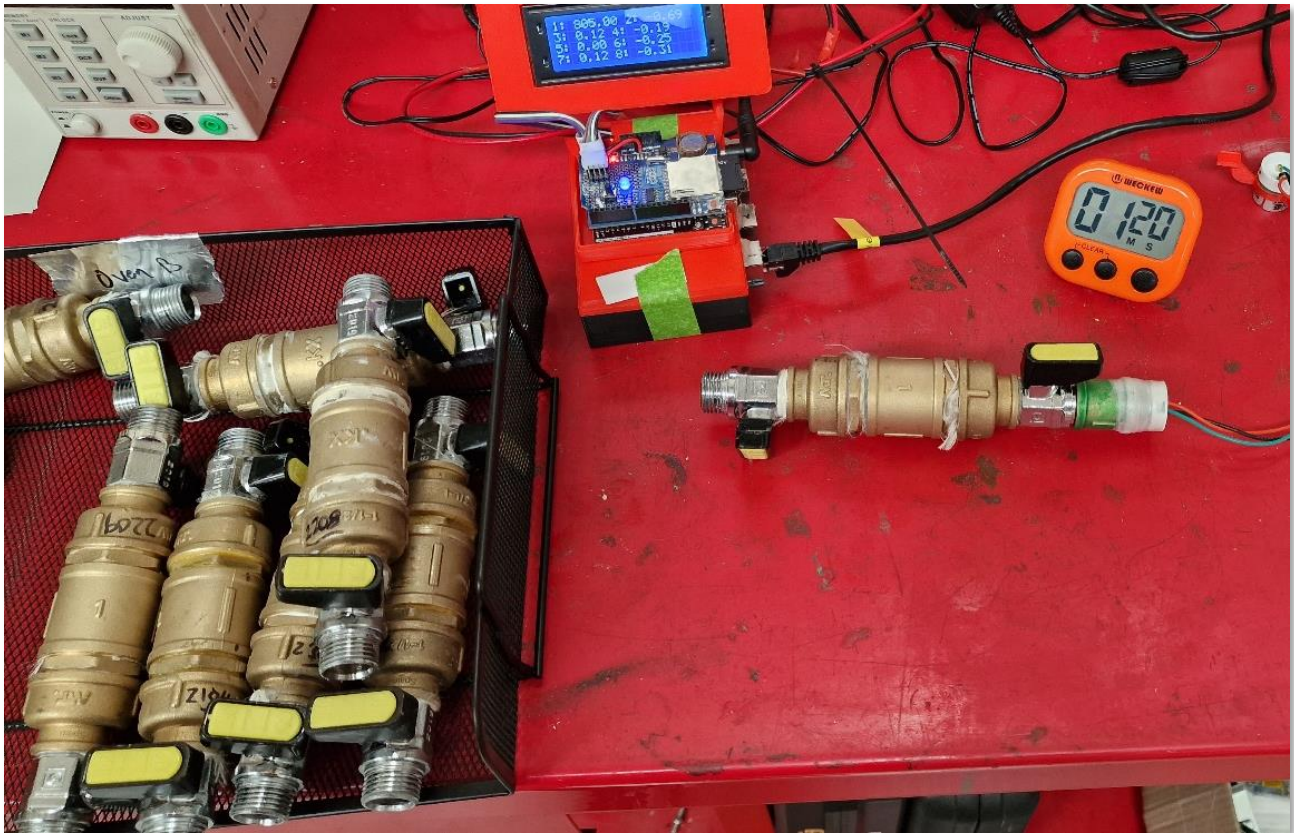


Figure 28: Hydrogen concentration test setup for week 1 tests

7.3.2 Retaining hydrogen

As noted, it quickly became apparent from the concentration tests that air was getting into the strings. This was a concern for the validity of the long term testing, as the Arrhenius model relies on the sealant being in contact with hydrogen. A regular purging of the leakiest strings was introduced, but this was not sustainable. A total of 12 options and variations were trialled in an attempt to reduce or prevent the ingress of air. Options included a variety of self-amalgamating tapes and combinations for both gas and water. These were leak tested at low pressure. A Testo 316-2 hydrogen gas detector was used to estimate relative differences in how much hydrogen was able to escape through each option.

None of the options prevented the escape of hydrogen at low pressure, and were therefore deemed to not be sealing sufficiently to prevent the exchange of gases. The most effective solution was using vacuum putty over aluminium tape. Ultimately the option used was a paper liner cut to fit between the shoulders of two joints, to protect the joint and any protruding fibres. This was covered with aluminium insulating tape sealed to the shoulder of the joint on both sides. Test pieces with particularly large leaks or a high rate of hydrogen concentration decay were also covered with vacuum putty. This is shown in Figure 29 on string 2203. The improvement in concentration retention when this was applied is clear in Figure 30.



Figure 29: Temporary external wrapping on leaky string 2203

Regular purging back to hydrogen was still required, but less frequently. This step added considerable time to each test, so was only done for strings with the largest leaks.

7.3.3 Exploring air ingress

Due to the extra time taken to carry out temporary external wrapping of strings, a series of experiments were carried out to determine which were the strings most in need. This also provides useful information regarding the air ingress phenomenon, but the data was only intended to be indicative.

To carry out these experiments, the strings were purged to 100% hydrogen, and the concentration measured at varying intervals. The concentration map for the strings with the highest leak rates at the time are shown in Figure 30. The trace shows the measured hydrogen at the point of test, not the hydrogen contained. Immediately after each test the string was purged back to 100% hydrogen. The sudden improvement in the results for strings such as 2301, 2203 and 6203-1 is due to the external wrapping process.

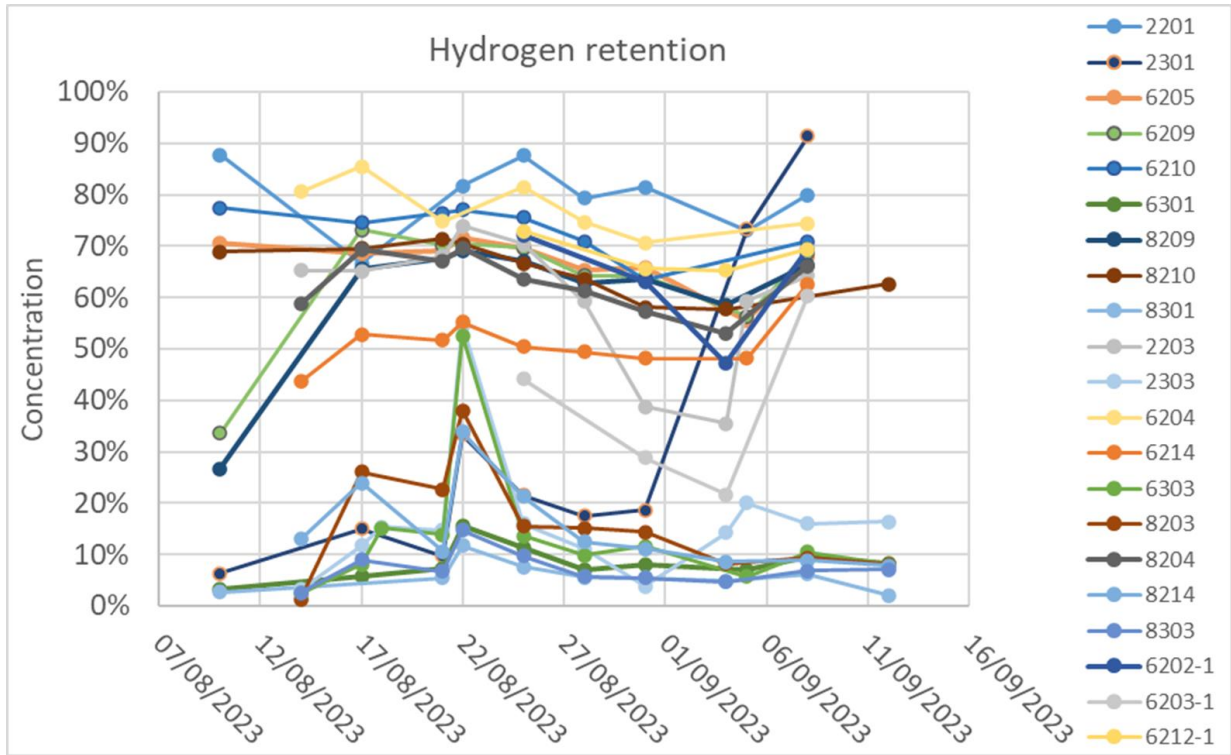


Figure 30: Concentration tests for the leakiest strings

A rate of percentage concentration loss per day was calculated, and seven strings stood out as having lower retention, especially when the concentration test was closer in time to the purge. The results for these are shown separately in Figure 31. These strings were identified as requiring regular purging.

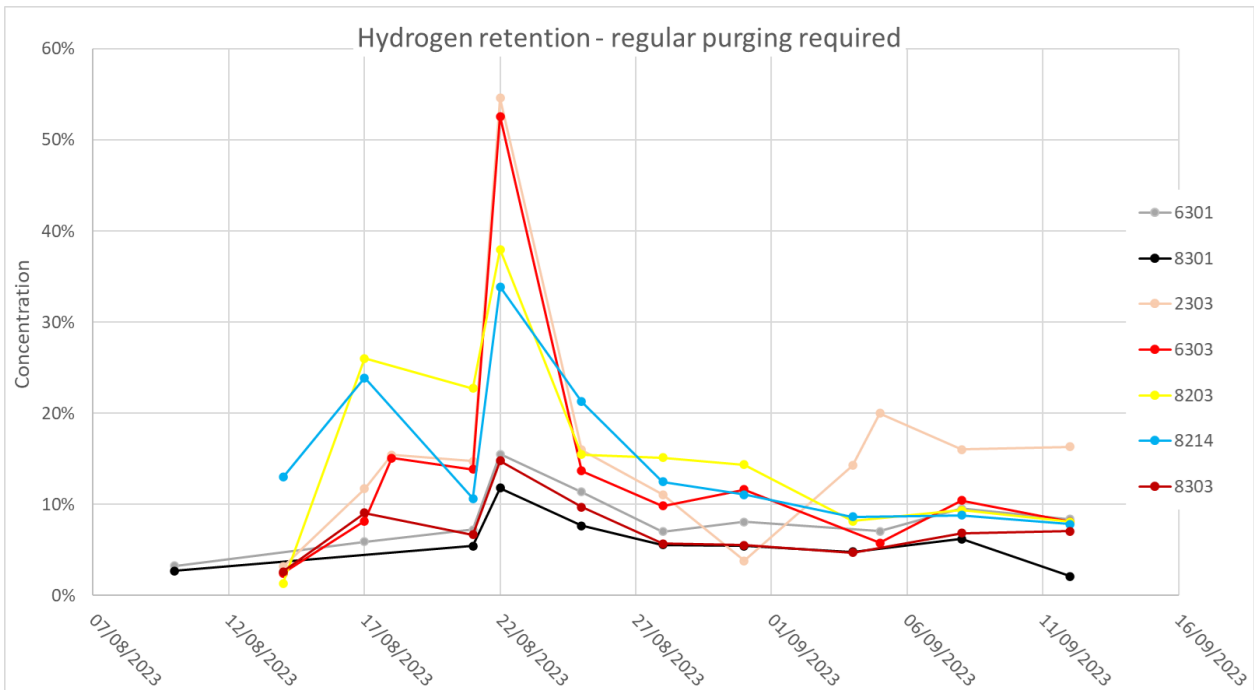


Figure 31: Strings with high air ingress

Of the seven worst strings, three retained a low concentration over a two day period. These were 6301, 8301 and 8303. All of the 303 (Thin PTFE Tape) strings were in the lowest 7 strings. Although they had external wrapping applied, they did not retain a significant concentration. 8203 and 8214 also did not retain concentration well, but the leak rate at the time of these tests was below the failure leak rate of 23 ml/min.

7.3.4 Concentration retention relative to leak rate

The concentration measured prior to each leak test was a good indicator of string leakage, but the correlation was not always good. As an example, the concentration measurements for the group 1 anaerobic sealants is provided in Figure 32.

String ref	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
2101	95%	98%	90%	95%	96%	84%	83%	84%	86%
6101	92%	91%	81%	87%	83%	81%	77%	79%	80%
8101	89%	78%	73%	78%	76%	64%	26%	66%	22%
2102	85%	94%	75%	55%	17%	8%	11%	12%	59%
6102	93%	92%	80%	86%	83%	81%	78%	79%	80%
8102	87%	73%	71%	69%	44%	33%	47%	40%	74%
2103	94%	95%	91%	94%	93%	83%	82%	84%	86%
6103	94%	96%	82%	88%	83%	81%	76%	78%	79%
8103	85%	78%	70%	73%	77%	70%	59%	77%	79%
2104	92%	93%	88%	91%	90%	81%	81%	81%	85%
6104	93%	90%	79%	79%	84%	80%	79%	79%	81%
8104	92%	75%	75%	60%	75%	59%	60%	57%	71%
2105	84%	87%	76%	85%	77%	77%	77%	79%	77%
6105	84%	84%	76%	78%	76%	76%	78%	79%	78%
8105	86%	85%	68%	74%	49%	4%	55%	1%	8%

Figure 32: Hydrogen concentration in anaerobic sealants prior to leak testing

The concentration was generally high, with notable low concentrations in strings 8101, 2102, 8102 and 8105. String 2102 has low concentrations for many of the tests, but this does not correlate to the leakage measured. The leak rate for 2102 is shown in Figure 33.

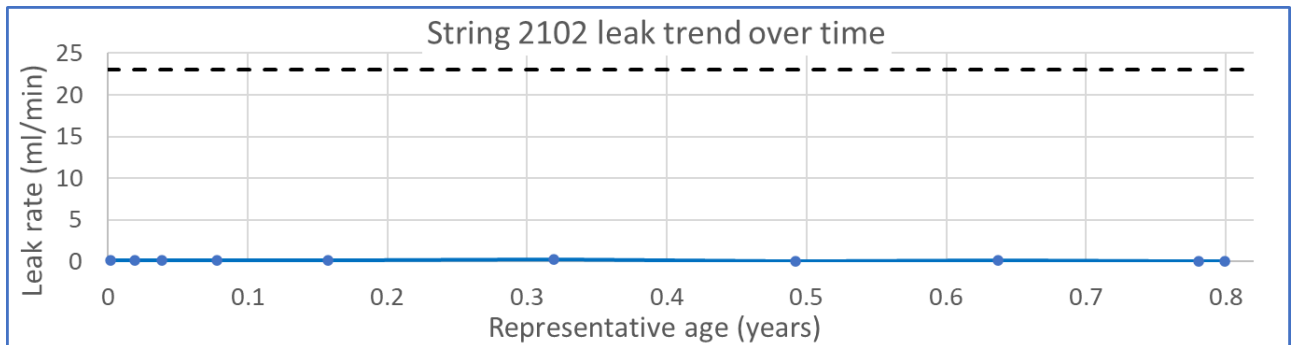


Figure 33: 2102 leak development over time

The leak rates for 8105 are shown in Figure 34, and represent a more expected profile when considering the concentrations measured.

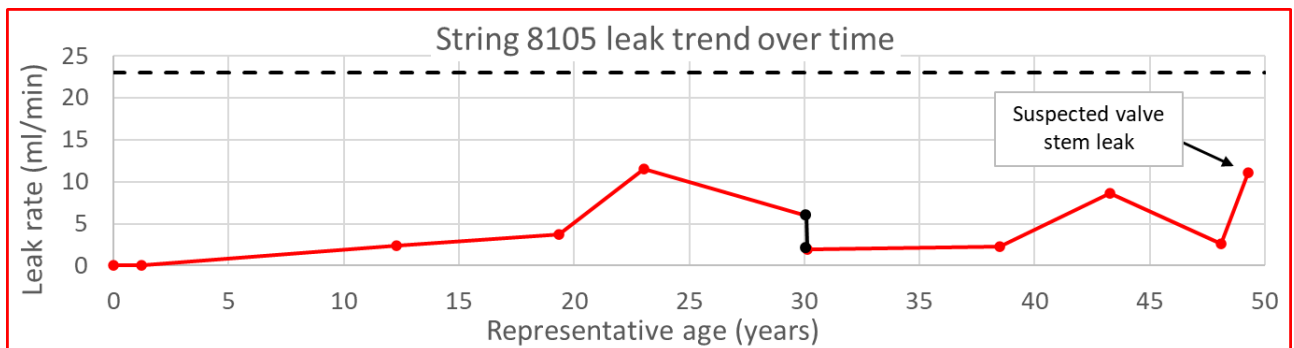


Figure 34: 8105 leak development over time

The issue of valve stems leaking appeared to be generally when the valves were open. Results such as that seen for 2102 may indicate valve stem leaks when the valve is closed. The mechanism for this is less likely however, due to the positioning of the seat seals relative to the stem leak path.

7.3.5 Comparison with methane

A series of further experiments were carried out comparing the rate of concentration decay between hydrogen and methane. This would highlight if the air ingress was a phenomenon particular to hydrogen. This was beyond the scope of this study, so could not be a detailed review.

As the 303 (thin PTFE tape) strings had leak rates substantially above the failure rate, and were not retaining concentration after a short period, they were due to be fully retired. Before undergoing the retirement process however, they were used for this investigation.

All three strings were purged and placed in the 20 °C oven. They were removed at intervals and the gas concentration measured. They were re-purged after each test, so the time shown is the total time from purge to concentration measurement for each variation. This was done with both hydrogen and methane in all three strings.

The results for the concentration decay over time are shown in Figure 35 for methane, and Figure 36 for hydrogen.

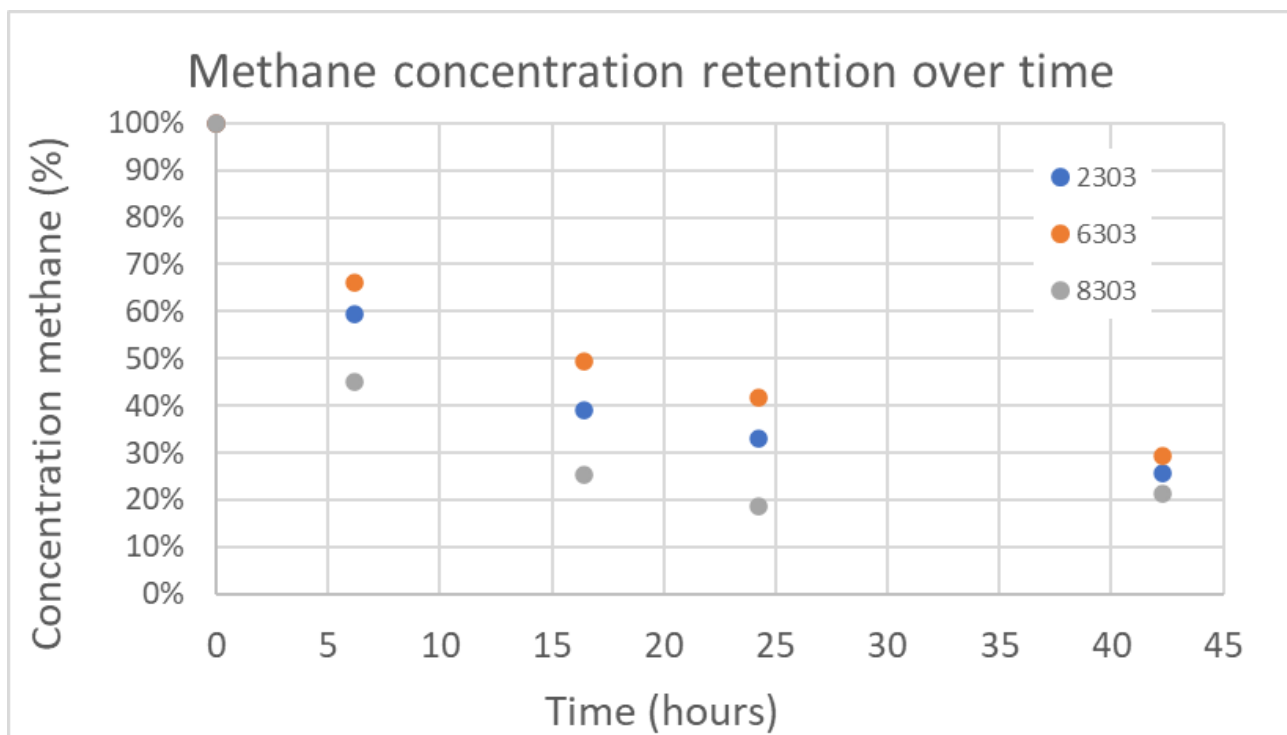


Figure 35: Methane concentration decay over time - graph

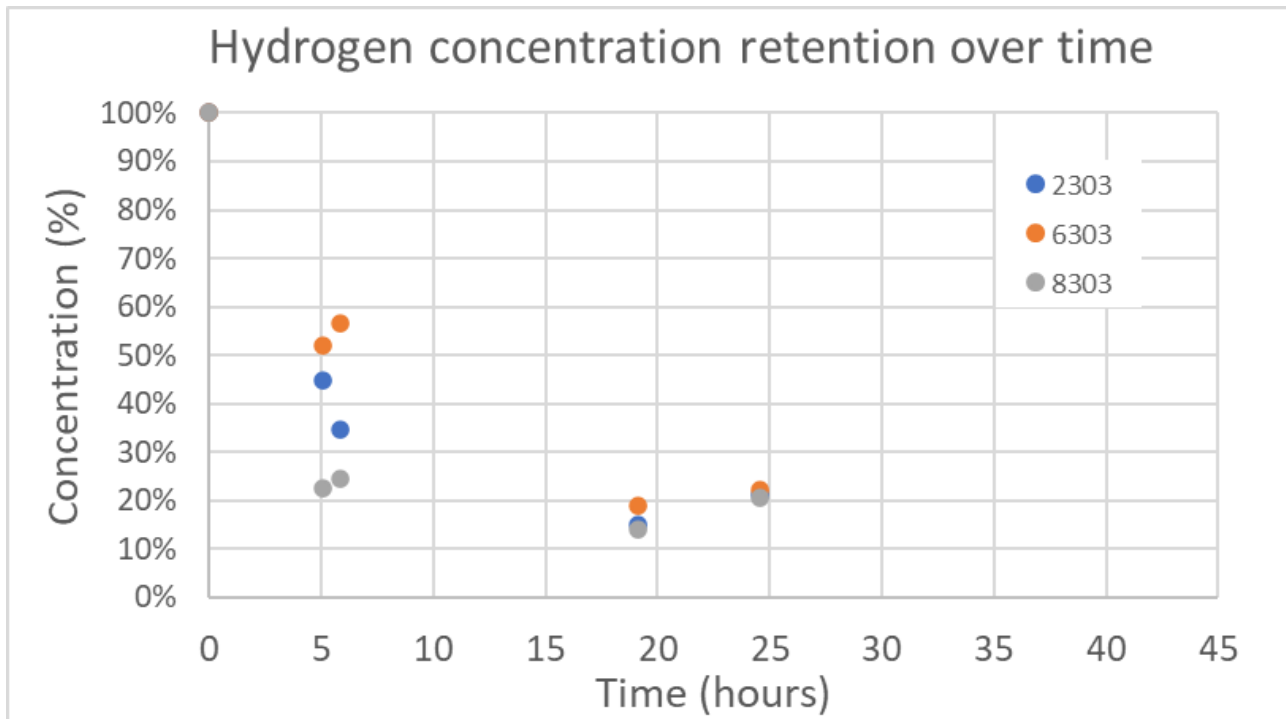


Figure 36: Hydrogen concentration decay over time - graph

The reduction in hydrogen concentration happens approximately three times as quickly as for methane. This finding is based on a very limited data set, but is an indication that the hydrogen concentration will fall quicker than methane.

It should be noted that the volume contained in these test samples is very small, which greatly amplifies the effect seen here. A more thorough investigation is required to determine if this phenomenon needs to be addressed.

7.4 Valve threaded connection leaks

As there were two valve connections for each string that were not part of the actual test data, it was prudent to minimise any leakage from these joints. The male thread on the valves was parallel, which does not seal optimally in a female parallel thread.

It was evident that the valve connection could not be sealed externally due to the shape of the connection, with valve stem protrusion, and was further complicated by the presence of extruded sealant materials. Figure 37 shows 2303 as an example.



Figure 37: Valve connection

The time taken to disassemble the valve connections, clean the threads, and reapply PTFE cord and compound proved inefficient during the valve replacement process for the initial strings, and the sealing results were mixed.

Bonded washers ('Dowty Washers') were therefore used, placed between the shoulder of the reducers and the shoulder of the valves. These are stainless steel rings with a slightly protruding NBR inner ring. The stainless steel ring prevents over-compression of the rubber seal.

The timing coincided with the realisation that the leakage attributed to the valve connection threads was often through the valve stem seals. New valves were required, so valves with a flat face that could be sealed with a bonded washer were procured. The new style of valve on string 8301 is shown in Figure 38.



Figure 38: Replacement valve with bonded washers

As the test programme progressed, identifying where leaks were coming from was incorporated as standard, and it was noted that some of the replaced valves began to leak from the threads again. This was the case for valve connections sealed with PTFE cord and compound, and bonded washers.

The bonded washers loosened and required retightening. This did not appear to correspond to a decrease in concentration retained, indicating that the loosening (and leakage) occurred after removal from the ovens. The cause is believed to be thermal contraction during cooling, while stress relaxation of the rubber is a possible contributory factor.

Changing the valves was incorporated into the leak test process for any strings where leakage was above the failure rate. A further programme of work has been recommended, to investigate the suitability of standard gas rated valves. The results gathered here may be useful for that study. This is highlighted in section 9.3.

7.5 Valve stem leaks

During leak testing it became apparent that a significant proportion of the leakage was from the valves. This was initially believed to be only from the valve threaded connection, but as the precision of the locating method was improved, it became clear that gas was also escaping through the stem seals when the valve was open.

Any leakage from the valves was indistinguishable from the test piece leakage, and made those results inaccurate. Leakage through or from valves should be subtracted from the total leak rate, alongside the system leak rate.

37 valve replacements were made in total throughout the test programme. Valve replacements were only done on strings that demonstrated leakage above the failure rate, due to time constraints. The valve replacements and associated decrease in leak rate are included in the test cards.

7.5.1 Replacement valves

The original valves used were sold as 'gas valves' from a leading online gas merchant. They were cheap and datasheets were not readily available. The advertised specification stated suitability for 80 °C. The seats were listed as NBR, but no material was given for the stem seals.

A range of gas valves was purchased as replacements. The stated maximum operating temperature for these was 90°C. An additional requirement at this stage was a flat face to provide a sealing surface for bonded washers.

7.5.2 Causes for valve leakage

Upon removal from the string, there was no obvious indication for the cause of leakage, and it was assumed to be degradation of the corner between the seat and stem seals. When a ball valve is open the shape of the cavity leaves a very thin section of seal exposed.

Without further analysis of the data and potentially further experiments, it is not clear whether the cause was temperature, ageing, or hydrogen incompatibility. A more detailed study would be needed to determine which mechanism is occurring and whether it has any significance for hydrogen installations.

7.6 Method of retiring failed joints

The overall aim of this study was to determine whether any sealants developed issues from contact with hydrogen. The initial plan therefore only included an assessment of pass or fail, and did not include investigating how high leak flow rates would reach.

The proposed plan stated that once a string leaked beyond the agreed MPLR, the leaking joint would be identified, and 'retired' using an external sealing method. This method was successfully trialled prior to proposing this work. This process removed the leaking joint from the string, meaning the leak test only included the other joints. Subtracting the difference would demonstrate the leak rate of the retired joint.

7.6.1 Planned retirement method

The proposed method for retiring joints that leaked above the agreed MPLR was trialled prior to agreeing the experimental plan. To produce this external seal, vacuum putty was pushed into the throat of the leaking joint, and wrapped with self-amalgamating tape. This example provided in the project proposal is shown in Figure 39. This was successful at sealing a joint in an example string with a deliberately created leak.



Figure 39: 'Retired' joint example

The putty was used for the first joint retirements, but based on the initial leak prevention results, *self-amalgamating tape* was not deemed necessary. The retirement process highlighted that there would be an issue with any joints where there was any protruding cord or tape, and those where the sealant had left an oily residue on the exposed parts. Self-amalgamating sealing tape would not be suitable for these strings, as it would not be able to adhere or seal to the fitting surface.

The first string with joint retirements was 2303. As the decision had been made to retire the entire string, the joints were retired in order, from A to B. Subtracting the difference each time a joint was retired gives the leak rate from the retired joint. The results for retirement are discussed along with each string, but the results for string 2303 are shown in Figure 40 to demonstrate how the process works.

2303 Retirement leakage by joint

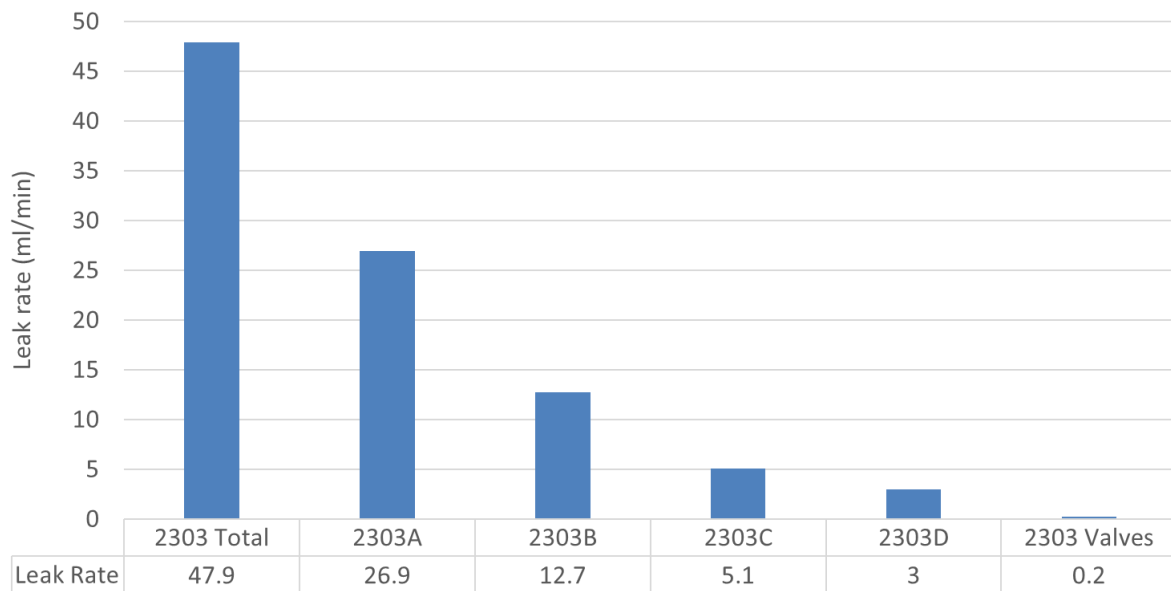


Figure 40: Joint by joint retirement of string 2303

6303 was retired next, and after sealing joints A and B the leakage reduced to 0.7 ml/min (0.00042 m³/hr). Joints C and D were therefore not retired, and the string was returned to the test programme, renamed '2303CDV' to reflect the remaining joints ('V' represents both valve connections).

Joints A, B and C were retired from string 8303, but once the ability to locate the leak location more precisely was gained, subsequent strings only had a single, targeted, joint retired.

7.6.2 Developments to the joint retirement method

All strings apart from 2303 were returned to their respective ovens and continued in the test programme. It was felt that potentially useful information could be gathered by attempting to continue the test for the remaining joints.

Subsequent leak tests however showed that the retirement carried out was not permanent. The leak rate increased for the strings, and locating the source indicated that the retired joint was at least partly responsible. The proportion of the leak that was from the already retired joints could not be determined.

The retirement method was reviewed and amended, and the decision made to carefully disassemble and remake the joints that had already been retired, using PTFE cord and compound. Care was taken to only use the flats on the joint being disassembled, to prevent affecting the remaining joints. Thin bladed adjustable spanners were used for this purpose. The barrel and 1" part of the reducer could be held loosely in a vice, with only the grip protrusions preventing rotation rather than clamping. This avoided compressing the fitting, which may have affected the non-retired joints. This process is demonstrated in Figure 41 for joint B of string 2202-3.



Figure 41: Loosening joint B of 2202-3

The retired joint was then wrapped with PTFE cord, leaving the existing compound in place on the threads. This step for joint B of string 2202-3 is shown in Figure 42.



Figure 42: Application of PTFE cord to retired joint B of string 2202-3

Finally, the previously retired joint was re-assembled. At this stage the string was renamed to represent only the remaining joints (including both valves). String 2202-3ACDV is shown in Figure 43.



Figure 43: String 2202-3 remade as string 2202-3ACDV

This method was successful initially, but again the leak rate increased over time. This rendered results for that string are invalid, as the proportion of the leak from the retired joint could not be determined.

Six strings had already undergone joint retirements. These were 2303, 6303, 8303, 6202-1, 6212-1, 2202-3, of which 2303 had been fully retired.

Due to the difficulties encountered while ensuring the tests for the rest of the string remained valid, other strings that developed leaks were not retired. Due to the air ingress challenges highlighted in section 7.3, the accuracy of the Arrhenius model was reduced for those strings with leaks. The leaky strings were purged on a regular basis, but the time requirements precluded purging with sufficient frequency to ensure hydrogen concentration remained high.

Using 1" bonded washers to re-seal retired joints was not attempted. Using these for the valve connections resulted in the need to replace the bonded washer after each temperature cycle, indicating that it was unlikely to be a suitable permanent solution for retirement. This was successful during the final leak test of any leaking strings however, as there was no requirement for ongoing sealing and temperature cycling.

7.6.3 Final leak test retirements

During the final round of tests, strings with leakage above 23 ml/min (0.00138 m³/hr) were targeted to determine where the leak was coming from. The difference in leak rate was subtracted, identifying how much of the leak was emanating from that joint. This is included on the test cards.

As an example, string 2201 was included for retirement of a joint, as the final leak rate recorded was 24.7 ml/min (0.001482 m³/hr). Prior to the retirement on the following day, the leak rate had decreased to 19.5 ml/min. Retiring joint D reduced the total leakage to 0.2 ml/min (0.00012 m³/hr), demonstrating the success of this method. String 2201 after the retirement of joint D is shown in Figure 44.



Figure 44: String 2201 after final leak test, with joint D retired

7.7 Summary of test steps

This flow chart aims to show the steps taken during the test programme. The stages changed over time, and this representation is the final process. As they were developed based on the findings, the early tests did not always consist of these steps.

Decision-making process at each step is shown in Figure 45, including the additional stages discussed in this chapter. This flow chart is a guide only, and has been applied retrospectively. The steps were developed during the test programme, so were not followed throughout or from the start. For example, the valve stems were not identified as a major source of leakage at the start of the test programme.

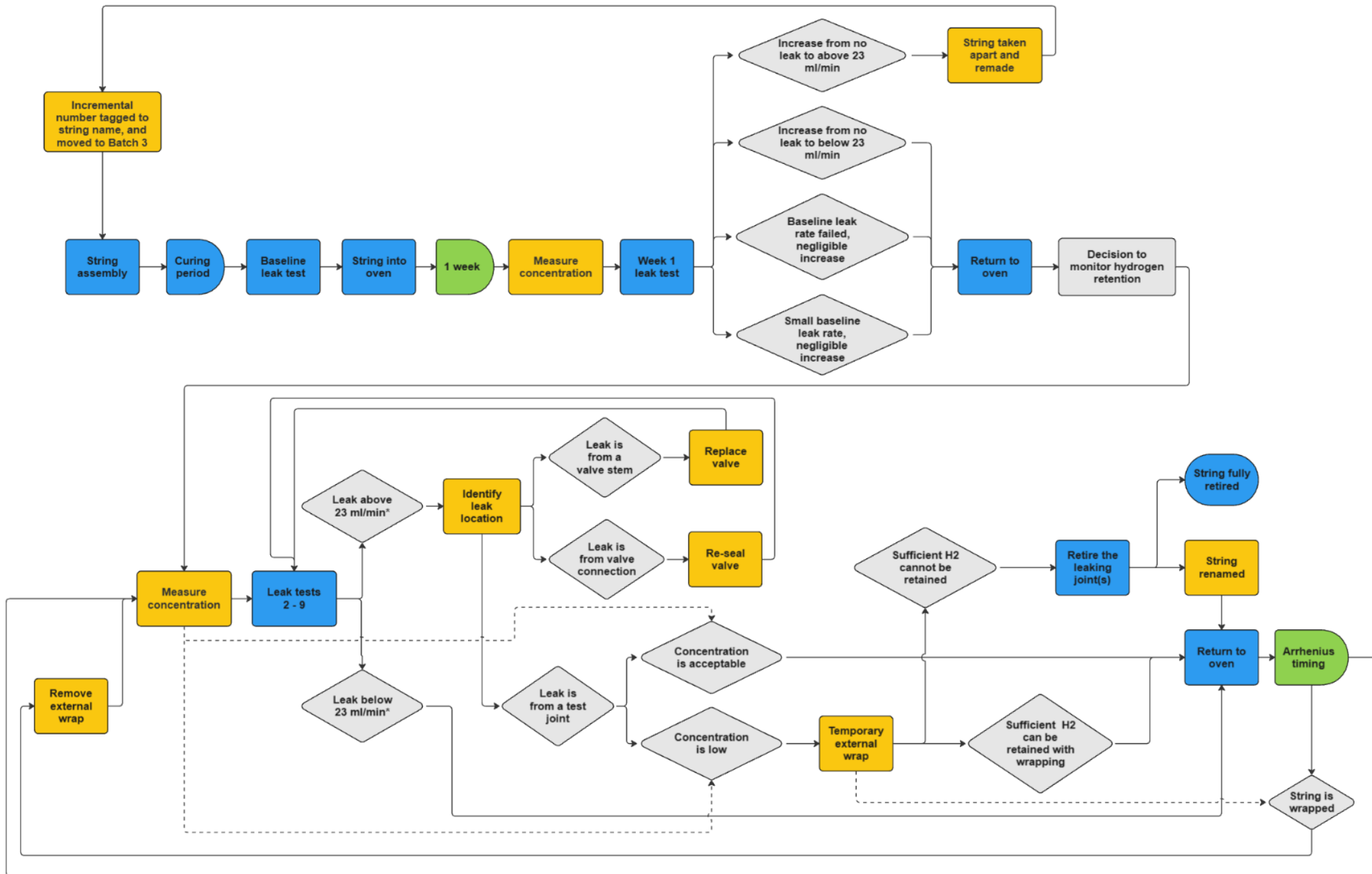


Figure 45: Flow chart of decision making and test procedures

8 Results

This chapter presents the leak test trend results for each string. An analysis is provided for sealant or sealant combinations that did not seal, or show particular effects. The majority of the strings remained leak free, and no discussion is required. All test cards and individual string leak trends are provided in Appendix A.

A section is also included to explore the validity of the Arrhenius method of artificial ageing, based on the challenges encountered during this project.

The results for all strings are given together initially, to provide the context of the results. Each group of sealant type is then discussed individually. For the purpose of reporting, the sealants are grouped into their categories within BS EN 751, while the non-hardening compounds in BS EN 751-2 are further split into those with and without PTFE cord as support. Although PTFE cords were included in group 2 during testing (as this was the standard stipulated by the manufacturers), PTFE cord alone (with no compound) is included with PTFE tape in the results section.

The individual results and graphs of leak trend over time are provided in full in Appendix A, with only some presented here to form part of the discussion. For all strings, blue represents the 20°C strings, with orange for the 60°C strings, and red for 80°C. Where valves have been replaced, the before and after data points are included as black spots. A black vertical line connects the two, representing the reduction in leak rate due to the valve replacement. A dashed horizontal line depicts the failure leak rate of 23 ml/min (0.00138 m³/hr). Before and after leak tests for retirements are shown by green spots, with the reduction in leak rate portrayed by a green vertical line.

When considering these results, it should be noted that the leak rate threshold of 23 ml/min (0.00138 m³/hr) is very conservative. This is based on the MPLR stipulated in IGE/UP/1, for the energy released by Natural Gas. The same volumetric leak flow rate is applied for hydrogen, and the transitional flow regime means the energy flow of hydrogen will be less than that of methane or Natural Gas for a given hole size. See section 6.1 for more detail and justification.

Note that the Y-axis is scaled depending on the leak rate. Where a valve leak renders the scale meaningless, the scale is generally reduced, to show the pertinent information.

All leak tests were carried out at a nominal 150 mbar unless otherwise stated. The test piece leakage was inferred by removing the system leakage from the total leakage. Due to slight variations in conditions, some of the leak rates are slightly below zero. These have been zeroed in the data processing to simplify the graphs.

The leak rates are given in ml/min as this was the recording format and is appropriate for the level of leakage and precision used when comparing joints. The summary of results also provides these leak rates in m³/hr, generally to 2 significant figures, to provide context within the gas industry.

If the leak rate was above 200 ml/min, a larger flow meter was used. This has lower precision, leading to lower precision in the results presented.

With the exception of string 8202, the leak rate results presented here are the baseline leakage when the string was assembled, the final leak rate, and the increase in leak rate. Due to the variability in leak rate over time for a single string, some of the differences are negative. This means occasionally the final string leak rate is less than the initial rate, indicating a reduction in leakage. This negative value has been retained.

The ratio of the increase in leak rate to the failure leak rate has been provided in the table of results for each sealant category. This is given as the result for the total string, and if an individual joint has been retired, the ratio for that joint is also given. Most of the results are based on the increase in leak rate, but this is not always appropriate if there is a baseline leak rate, or the remaining leak rate after joints were retired is high. These are therefore noted within the table. String 8202 in section 8.5 also refers to the increase to the peak leak rate, rather than the final leak rate.

Leak rates between 10% and 200% of the failure rate are highlighted in light orange, regardless of whether the test was considered a pass or fail. Leak rates more than double the failure rate is highlighted in dark

orange. This provides a visual indication of the severity of leakage. In the results tables, strings that had joint retirements during the test programme are highlighted in grey.

A chart has been included to show the concentration of hydrogen at each leak test. This is intended only to act as a guide and an indication of hydrogen retention. It is also occasionally useful for analysis and interpretation of the results. Where the concentration is consistently low, there is uncertainty about the amount of contact with hydrogen the sealant has had, though the strings were filled with hydrogen after each leak test.

8.1 Experimental limitations - Arrhenius application

The Arrhenius model is well known and proven, however the application of it is dependent on several factors. In this study, leak rate is used to monitor deterioration of sealants, and therefore infer chemical reactions. This relationship is likely to be complicated by what actually happens in a threaded joint when sealants change over time. Unless a sealant is completely removed, a long thin leak path remains, which will be physically clogged with sealant. A reduction in viscosity of a sealant may allow extrusion along the thread due to pressure, while if it dries this may allow cracks to develop. In either case, the leak rate will reach an asymptote as the sealant deterioration increases. Further deterioration of the sealant will not necessarily lead to a higher leak rate.

Additionally, the concentration of (in this case) hydrogen is assumed to be 100% for the model to be accurate. If the concentration is lower, the rate of chemical reactions with hydrogen will decrease.

To provide reassurance of the validity of the ageing model, the schedule was arranged to overlap some of the datapoints for different temperatures – at the expense of a narrower overall distribution. The concentration of hydrogen was also measured immediately prior to each leak test, as outlined in section 7.3.

The findings from these steps are discussed in the following chapters.

8.1.1 Scheduled test overlaps

Some overlapping tests between different temperatures were included in the schedule to provide some assurance regarding the validity of the accelerated ageing. These are circled in Figure 46. Note that the x-axis portrays the total test number, not that for each string or temperature.

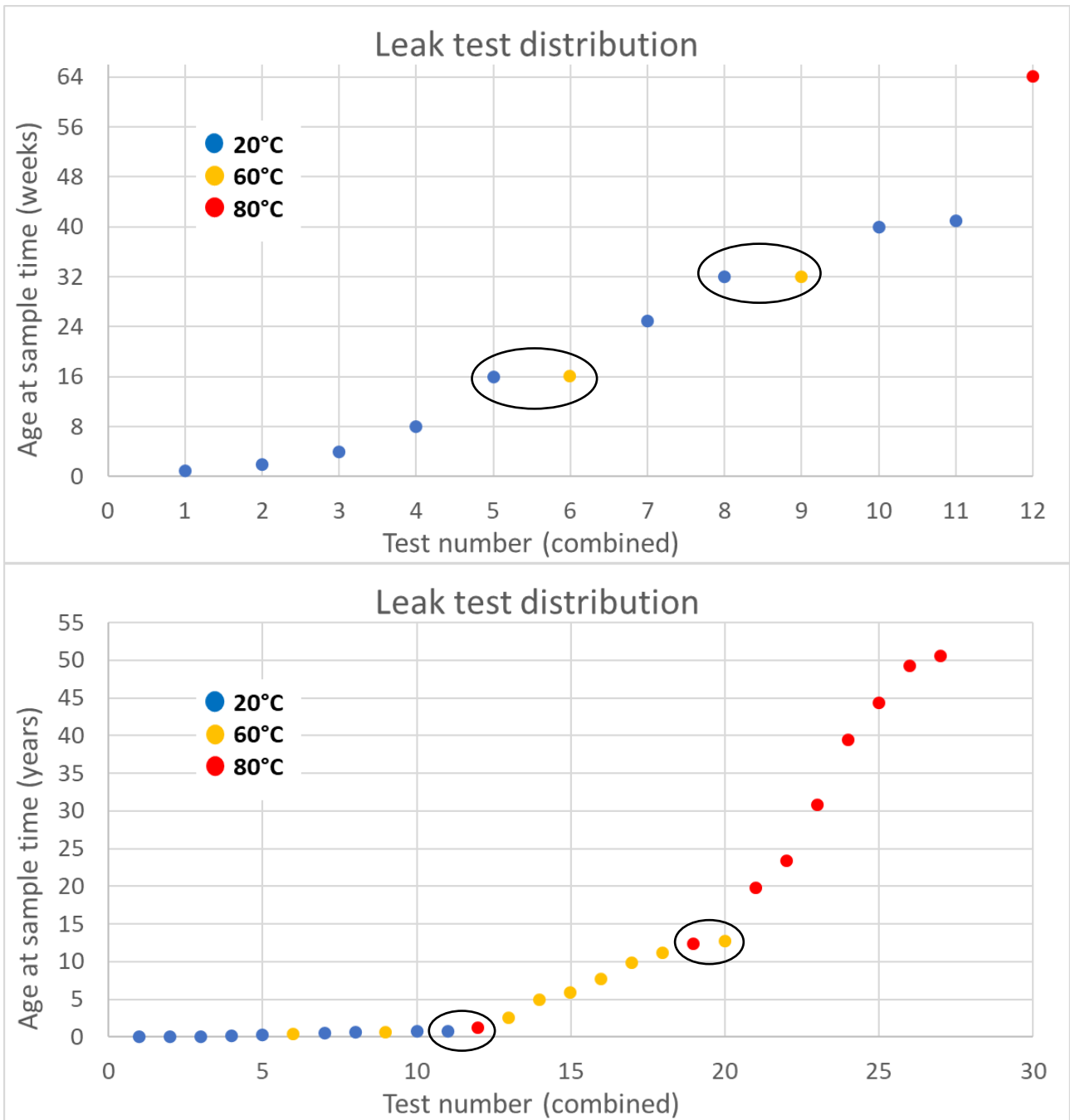


Figure 46: Approximate duplication of test dates

All leak rates are plotted against the total age in Figure 47. There is no indication of leak rate increase over the test programme. This does not show the trend of individual test pieces. The results are not included for any string after a retirement has taken place. This may account for the apparent decrease in the higher leak rates.

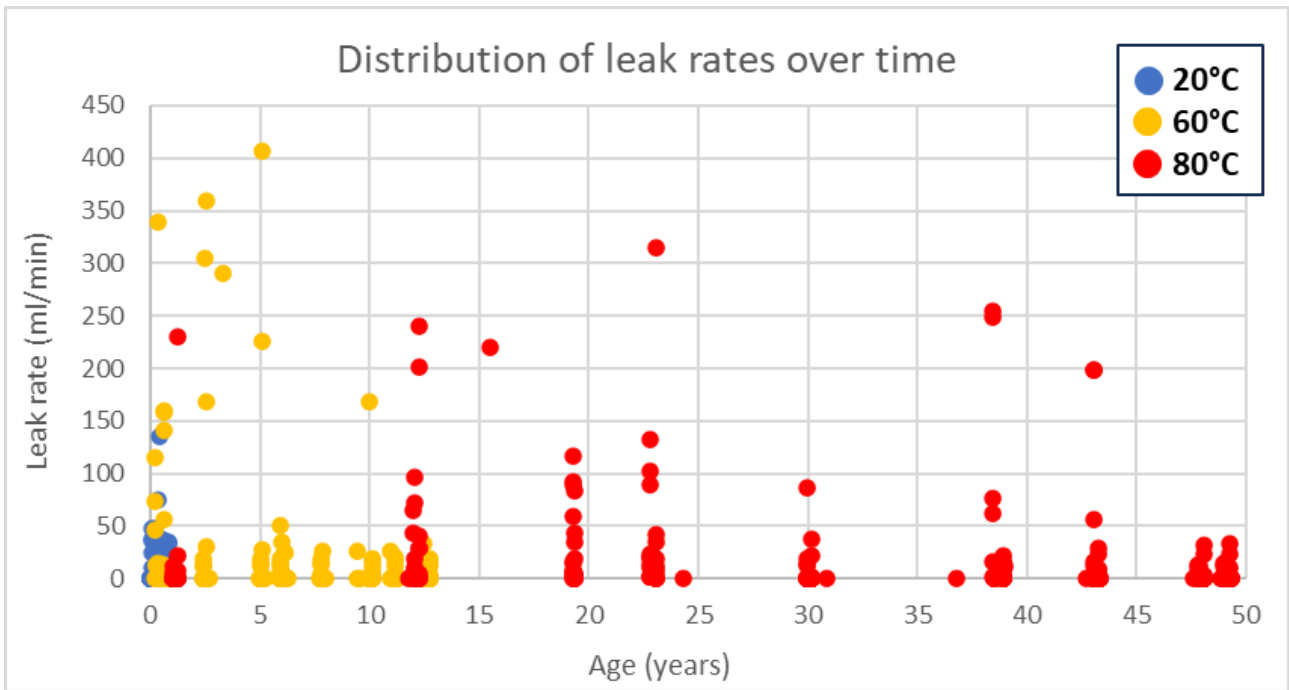


Figure 47: Distribution of leak rates over time - all data

When this is scaled to show the early test results (Figure 48), there is potentially an indication of an upward trend with increasing age.

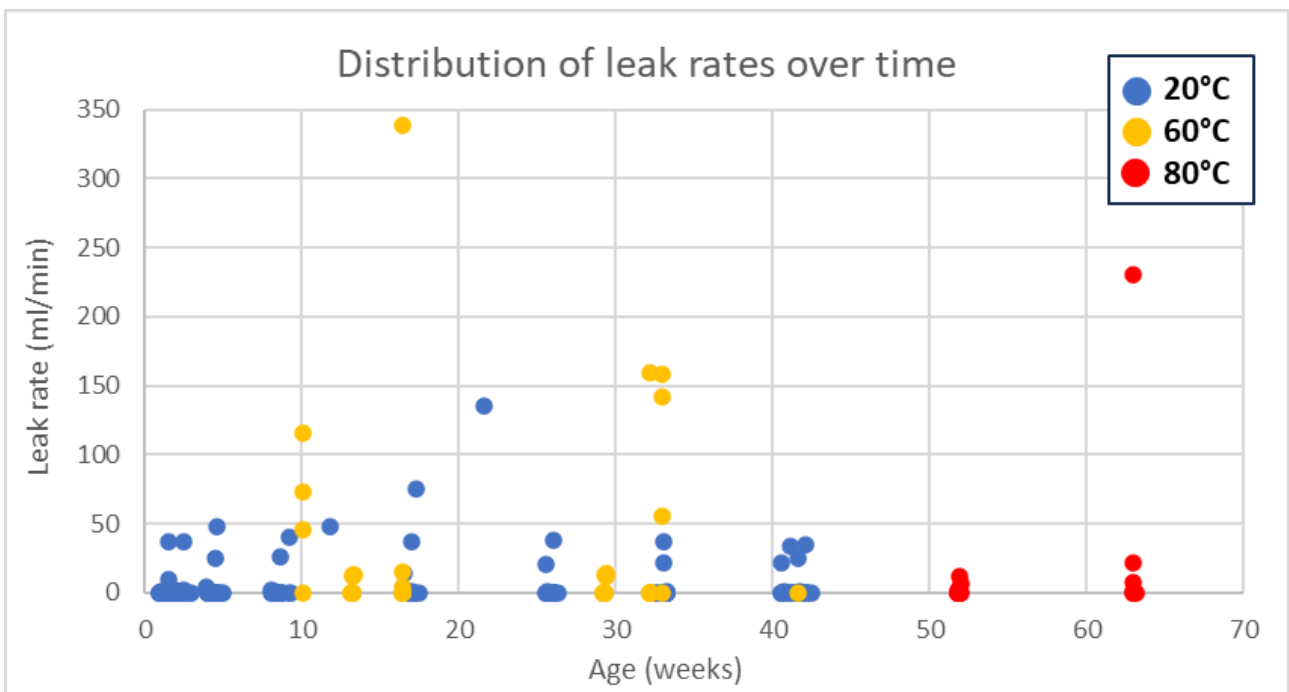


Figure 48: Scatter of leak rate development over time

The dataset is too small relative to the deviation to draw a firm conclusion however, and cannot prove the accuracy of the Arrhenius chemical ageing in this case.

8.1.2 Concentration of hydrogen

The Arrhenius model relies on contact with hydrogen. Hydrogen concentration tests were included immediately prior to each leak test, to provide an indication of the validity of the model. This provides reassurance that the majority of the strings without leaks remained in contact with hydrogen throughout the test programme, but some of the strings had a low concentration and therefore the ageing calculation is less accurate. Figure 49 shows how the concentration retained varies by temperature.

Temp.	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
20°C	95%	98%	90%	95%	96%	84%	83%	84%	86%
	85%	94%	75%	55%	17%	8%	11%	12%	59%
	94%	95%	91%	94%	93%	83%	82%	84%	86%
	92%	93%	88%	91%	90%	81%	81%	81%	85%
	84%	87%	76%	85%	77%	77%	77%	79%	77%
	88%	67%	81%	86%	86%	60%	63%	66%	76%
	89%	74%	88%	90%	69%				
						71%	84%	76%	76%
	65%	68%	35%	81%	14%	4%	3%	3%	12%
	89%	87%	80%	90%	81%	79%	80%	81%	82%
	86%	84%	70%	89%	76%	72%	82%	84%	84%
	92%	94%	91%	92%	93%	82%	82%	82%	86%
	92%	93%	89%	91%	92%	81%	81%	83%	85%
	94%	95%	81%	94%	87%	81%	83%	85%	82%
	89%	91%	75%	89%	73%	74%	75%	76%	79%
	90%	89%	80%	92%	81%	80%	83%	83%	82%
	97%	97%	88%	89%	87%	77%	75%	76%	80%
	88%	89%	78%	89%	84%	77%	80%	80%	80%
	94%	95%	81%	94%	87%	81%	83%	85%	82%
	89%	91%	79%	92%	85%	80%	82%	84%	82%
	98%	92%	101%	102%	99%	99%	102%	97%	94%
	95%	94%	89%	92%	90%	79%	80%	80%	86%
	97%	92%	88%	91%	89%	69%	71%	72%	69%
	6%	15%	19%	45%	16%	3%	3%	4%	13%
	92%	94%	78%	93%	80%	82%	81%	80%	83%
	3%	15%	14%	35%					
	93%	93%	79%	93%	82%	81%	83%	84%	83%

Temp.	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
60°C	92%	91%	81%	87%	83%	81%	77%	79%	80%
	93%	92%	80%	86%	83%	81%	78%	79%	80%
	94%	96%	82%	88%	83%	81%	76%	78%	79%
	93%	90%	79%	79%	84%	80%	79%	79%	81%
	84%	84%	76%	78%	76%	76%	78%	79%	78%
	87%	70%	79%	76%	78%	61%	0%	60%	60%
	72%	47%	77%	61%					
					70%	62%	56%	62%	64%
	44%	22%	56%	34%	1%	1%	0%	1%	0%
	81%	75%	75%	71%	73%	71%	70%	71%	74%
	95%	95%	81%	72%	79%	75%	73%	78%	79%
	94%	92%	83%	84%	82%	80%	78%	78%	81%
	77%	75%	76%	76%	67%	61%	59%	59%	63%
	73%	65%	75%	66%					
						59%	56%	6%	12%
	44%	52%	69%	43%	39%	7%	5%	23%	11%
	89%	88%	83%	82%	79%	79%	81%	81%	81%
	93%	94%	83%	85%	82%	81%	81%	79%	82%
	91%	89%	85%	83%	80%	82%	82%	81%	81%
	91%	87%	80%	70%	74%	84%	79%	81%	81%
	99%	92%	88%	98%	99%	97%	95%	99%	94%
	71%	68%	68%	59%	49%	23%	28%	42%	38%
	34%	73%	56%	55%	47%	6%	4%	14%	10%
	3%	6%	9%	7%	9%	1%	8%	1%	1%
	93%	92%	86%	84%	83%	80%	82%	84%	82%
	2%	14%	57%						
				6%	6%	1%	10%	1%	1%
	90%	88%	71%	35%	50%	37%	30%	38%	36%
80°C	89%	78%	73%	78%	76%	64%	26%	66%	22%
	87%	73%	71%	69%	44%	33%	47%	40%	74%
	85%	78%	70%	73%	77%	70%	59%	77%	79%
	92%	75%	75%	60%	75%	59%	60%	57%	71%
	86%	85%	68%	74%	49%	4%	55%	1%	8%
	95%	77%	75%	67%	77%	66%	73%	74%	78%
	91%	59%	70%	63%	34%	73%	55%	59%	73%
	1%	15%	3%	4%	1%	0%	1%	1%	5%
	59%	70%	63%	56%	53%	50%	55%	57%	56%
	71%	61%	34%	37%	26%	5%	16%	25%	38%
	64%	63%	63%	53%	53%	51%	50%	55%	39%
	69%	65%	2%	16%	0%	0%	3%	5%	14%
	93%	2%	61%	57%	74%	72%	77%	77%	78%
	13%	15%	3%	3%	0%	0%	0%	0%	1%
	91%	83%	71%	72%	68%	68%	76%	76%	80%
	93%	61%	74%	71%	0%	62%	76%	77%	70%
	90%	83%	70%	74%	75%	69%	63%	79%	76%
	90%	76%	75%	71%	72%	71%	77%	77%	78%
	100%	98%	97%	100%	88%	99%	93%	100%	99%
	92%	75%	77%	76%	78%	75%	77%	79%	82%
	27%	63%	39%	36%	15%	2%	10%	9%	38%
	3%	5%	4%	3%	1%	0%	0%	0%	3%
	88%	81%	66%	70%	54%	39%	50%	52%	52%
	3%	25%							
			3%	4%	1%	0%	1%	1%	6%
	91%	83%	68%	74%	71%	54%	59%	60%	50%

Figure 49: Comparison of concentration retained by temperature

Both PTFE only datasets (301 and 303) have been removed from all charts for this comparison. Retention was very low for these at all three temperatures, adding confusion to the overall result.

Some of the strings with higher leak rates were temporarily sealed externally, in an attempt to retain a higher hydrogen concentration. The strings that still showed low concentration generally were also purged to 100% hydrogen more regularly than the leak test schedule. The timings for these was not recorded, and is not adjusted for in this data.

Comparing the concentration retained across the three temperatures shows that overall as the temperature increased, the concentration retained was lower. This indicates that air ingress does not correlate with the increase in leak rate, which was greatest in the 60°C strings.

By the end of the test programme, the pattern concentration retained in the 60°C and 80°C strings is similar, but at the start the 80°C strings show lower concentration retained.

It is possible that less hydrogen being retained in the 80°C strings is the cause for leaks not developing as large as in the 60°C strings.

8.1.3 Arrhenius validity

Overall, the confidence of the Arrhenius model with this dataset is low due to the variation in air ingress between different temperatures. There was 100% hydrogen concentration following each leak test however, and regular purging for the leakiest strings. This means there was the opportunity for chemical reactions with hydrogen to take place, but potentially at a different rate from if there was 100% hydrogen contact throughout. There is not a good correlation between age and leak rate, which could be due to a lack of hydrogen retention, or because of the maximum leak rate that a deteriorating sealant would allow.

8.2 Overall results

A plot of the increase in leak rate at the final test for each string is given in Figure 50. The exception is 8202, where there was a particularly high peak during the testing which had reduced to nothing by the final test. For this string the peak is included. Where a string had joints retired the point at which that took place is included, as is the 'replacement' string.

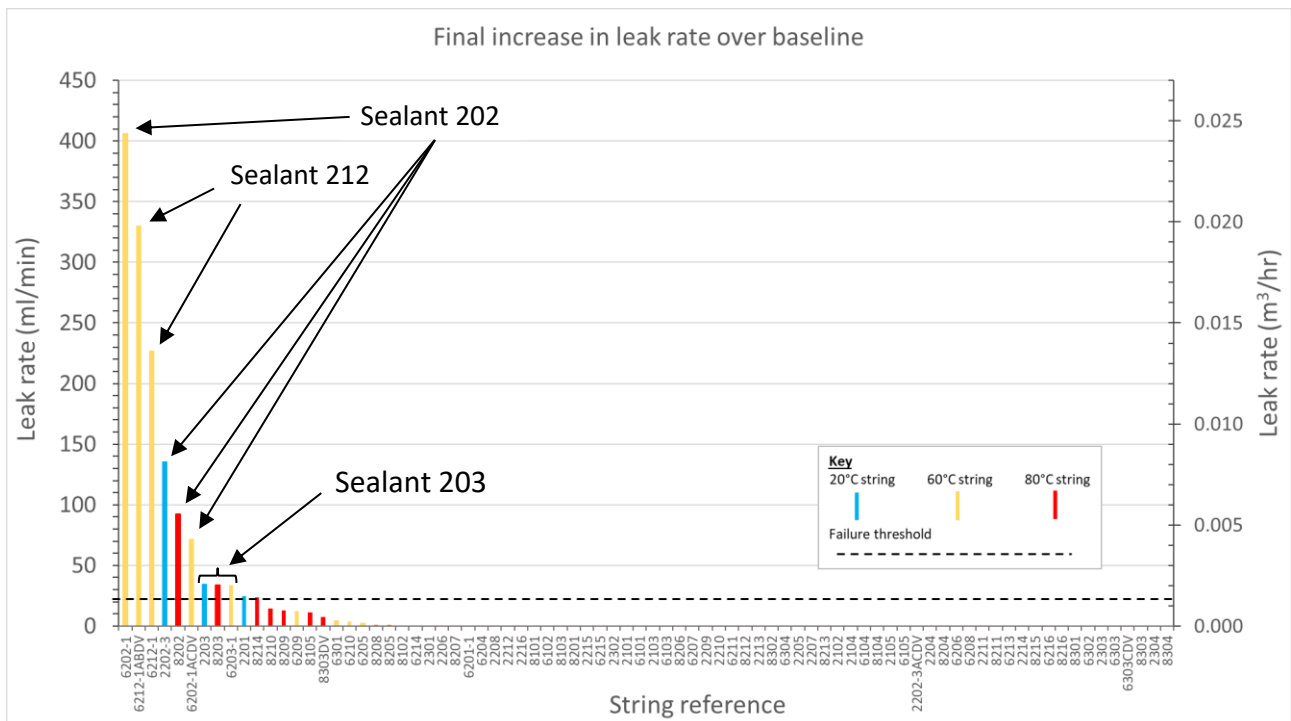


Figure 50: Final leak rates

In total, five sealants and a total of eleven strings had an increase in leakage above the failure rate. This includes 6212-1 and 6202 that had a joint retired and subsequently developed a leak from the remaining joints. Three others had a leak rate above the failure rate from the start, but did not deteriorate (therefore not apparent in Figure 50). Three sealants were accountable for the nine highest increases in leak rate. After the initially leaking joint in string 6212-1 was retired, two other joints also began leaking. The 20°C and 80°C strings for this sealant did not develop leakage however.

8.3 Anaerobic jointing compounds (BS EN 751-1)

8.3.1 Overview of results

The anaerobic sealants all passed the test, with less than 23 ml/min (0.00138 m³/hr) leakage. The baseline leak rates of each string was essentially zero. The overall results considering the increase in leak rate from baseline to final test is shown in Figure 51.

String	Temp.	Baseline leak rate (ml/min)	Final leak rate (ml/min)	Increase in leak rate (ml/min)	Pass/Fail	Ratio to failure rate
2101	20 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
6101	60 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
8101	80 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
2102	20 °C	0.1	0.0	-0.1	Pass	Total (increase): 0.0
6102	60 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
8102	80 °C	0.1	0.7	0.6	Pass	Total (increase): 0.0
2103	20 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
6103	60 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
8103	80 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
2104	20 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
6104	60 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
8104	80 °C	0.1	0.0	-0.1	Pass	Total (increase): 0.0
2105	20 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
6105	60 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
8105	80 °C	0.0	11.1	11.1	Pass	Total (increase): 0.5

Figure 51: Final leak rates for anaerobic sealants

There was no leakage observed from any of the strings during the test programme. When the test cards in Appendix A are reviewed, it is clear that any leakage measured was attributed to the valves. The only potential exception is 8105, discussed below.

8.3.2 Analysis of leaking strings

String 8105 developed a higher leak rate than the other anaerobic sealants. The same sealant at 20°C and 60°C showed no sign of leakage. The leak trend over time for these strings is shown in Figure 52 and Figure 53.

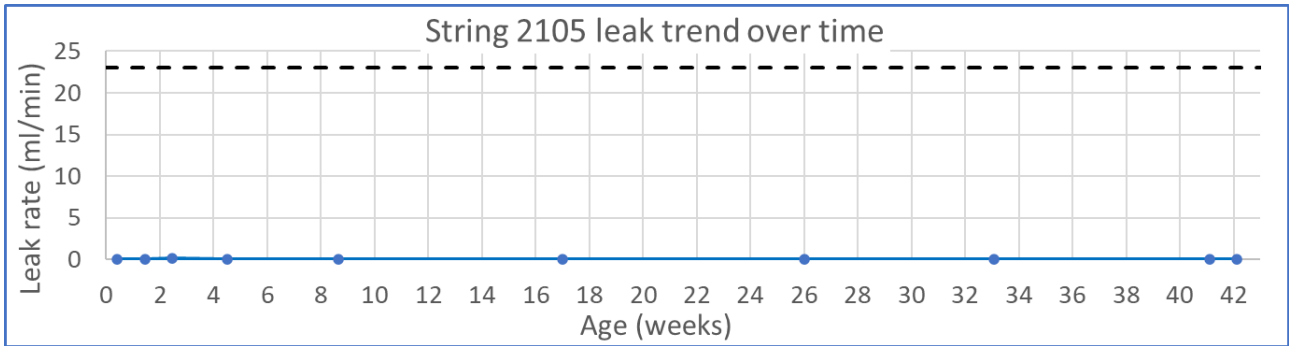


Figure 52: 2105 leak development over time

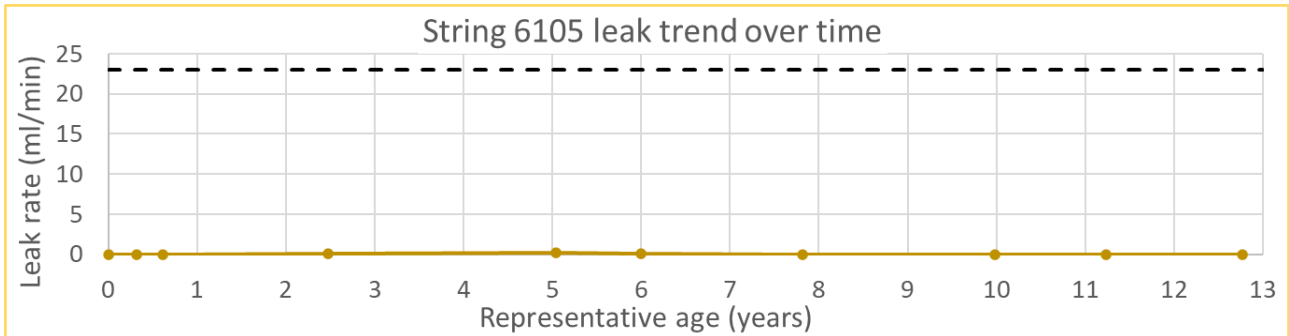


Figure 53: 6105 leak development over time

Figure 54 shows the leak rate trend of string 8105 over time. After test 4 this was identified as predominantly being from the stem of Valve 1.

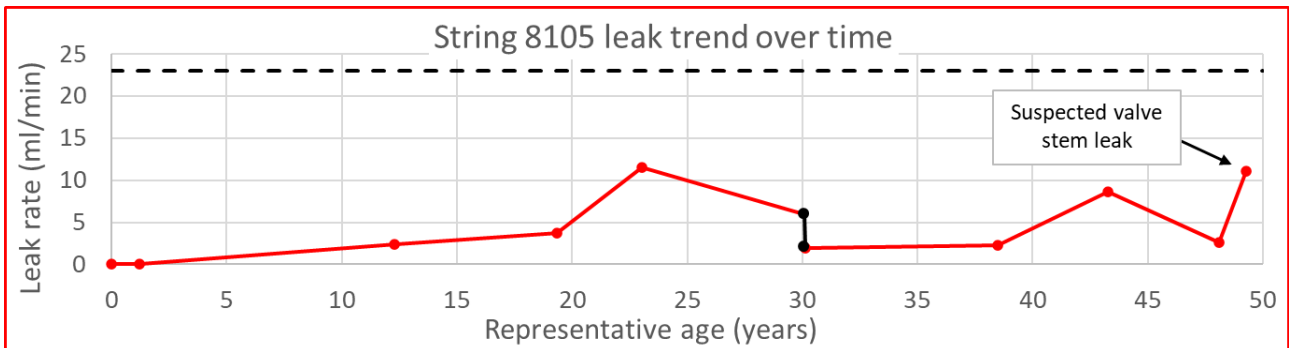


Figure 54: 8105 leak development over time

Valve 1 was replaced during test 5, reducing the leak rate from 11.5 ml/min (0.00069 m³/hr) to 1.9 ml/min (0.00011 m³/hr). At the final test, valve 1 was identified as having a stem leakage again, which was expected to account for the leak rate recorded. This was not replaced as the leak rate was below the failure rate.

When valve 1 was replaced between tests 5 and 6, the leakage reduced to 2.1 ml/min, which was then recorded as 1.9 ml/min in test 6. As the overall leakage did not reduce to zero, this could indicate a small leak in the region of 2-3 ml/min from a test joint.

8.3.3 Hydrogen concentration prior to leak test

The hydrogen concentration recorded immediately prior to each scheduled leak test is given in Figure 55.

String ref	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
2101	95%	98%	90%	95%	96%	84%	83%	84%	86%
6101	92%	91%	81%	87%	83%	81%	77%	79%	80%
8101	89%	78%	73%	78%	76%	64%	26%	66%	22%
2102	85%	94%	75%	55%	17%	8%	11%	12%	59%
6102	93%	92%	80%	86%	83%	81%	78%	79%	80%
8102	87%	73%	71%	69%	44%	33%	47%	40%	74%
2103	94%	95%	91%	94%	93%	83%	82%	84%	86%
6103	94%	96%	82%	88%	83%	81%	76%	78%	79%
8103	85%	78%	70%	73%	77%	70%	59%	77%	79%
2104	92%	93%	88%	91%	90%	81%	81%	81%	85%
6104	93%	90%	79%	79%	84%	80%	79%	79%	81%
8104	92%	75%	75%	60%	75%	59%	60%	57%	71%
2105	84%	87%	76%	85%	77%	77%	77%	79%	77%
6105	84%	84%	76%	78%	76%	76%	78%	79%	78%
8105	86%	85%	68%	74%	49%	4%	55%	1%	8%

Figure 55: Hydrogen retention for BS EN 751-1 – Anaerobic Sealants

The concentration was generally high, with notable low concentrations in strings 8101, 2102, 8102 and 8105.

8.4 Non-hardening jointing compounds with support material (BS EN 751-2)

The non-hardening jointing compounds complying with BS EN 751-2 were split into two categories. The majority were assembled with only the compound, and three sets used PTFE cord to support the compound. The hemp and compound strings (216) have been included in this group.

8.4.1 Overview of results

The initial baseline leak rate, final leak rate and the increase for non-hardening compounds with support material is presented in Figure 56.

String	Temp.	Baseline leak rate (ml/min)	Final leak rate (ml/min)	Increase in leak rate (ml/min)	Pass/Fail	Ratio to failure rate
2206	20 °C	0.1	0.3	0.2	Pass	Total (increase): 0.0
6206	60 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
8206	80 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
2211	20 °C	0.1	0.1	0.0	Pass	Total (increase): 0.0
6211	60 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
8211	80 °C	0.1	0.1	0.0	Pass	Total (increase): 0.0
2213	20 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
6213	60 °C	0.1	0.0	-0.1	Pass	Total (increase): 0.0
8213	80 °C	0.2	0.2	0.0	Pass	Total (increase): 0.0
2216	20 °C	0.0	0.2	0.2	Pass	Total (increase): 0.0
6216	60 °C	0.1	0.0	-0.1	Pass	Total (increase): 0.0
8216	80 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0

Figure 56: Final leak rates for non-hardening jointing compounds and PTFE cord support

There was no leakage from the non-hardening compounds when support material was used. This includes hemp and compound, which is no longer permitted on joints of this type.

8.4.2 Hydrogen concentration prior to leak test

The hydrogen concentration recorded immediately prior to each scheduled leak test is given in Figure 57.

String ref	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
2206	97%	97%	88%	89%	87%	77%	75%	76%	80%
6206	93%	94%	83%	85%	82%	81%	81%	79%	82%
8206	93%	61%	74%	71%	0%	62%	76%	77%	70%
2211	88%	89%	78%	89%	84%	77%	80%	80%	80%
6211	91%	89%	85%	83%	80%	82%	82%	81%	81%
8211	90%	83%	70%	74%	75%	69%	63%	79%	76%
2212	94%	95%	81%	94%	87%	81%	83%	85%	82%
2213	89%	91%	79%	92%	85%	80%	82%	84%	82%
6213	91%	87%	80%	70%	74%	84%	79%	81%	81%
8213	90%	76%	75%	71%	72%	71%	77%	77%	78%
2216	98%	92%	101%	102%	99%	99%	102%	97%	94%
6216	99%	92%	88%	98%	99%	97%	95%	99%	94%
8216	100%	98%	97%	100%	88%	99%	93%	100%	99%

Figure 57: Hydrogen retention for BS EN 751-2 – Non-hardening jointing compounds with support material

Note that the secondary calibration based on test setup was not carried out separately for the hemp and putty strings, so these appear slightly higher than reality. There is not expected to be a significant difference, and they retained hydrogen concentration well.

The concentration is good for all strings, with the exception of string 8206 in test 5. The leak trend of 8206 is shown in Figure 58.

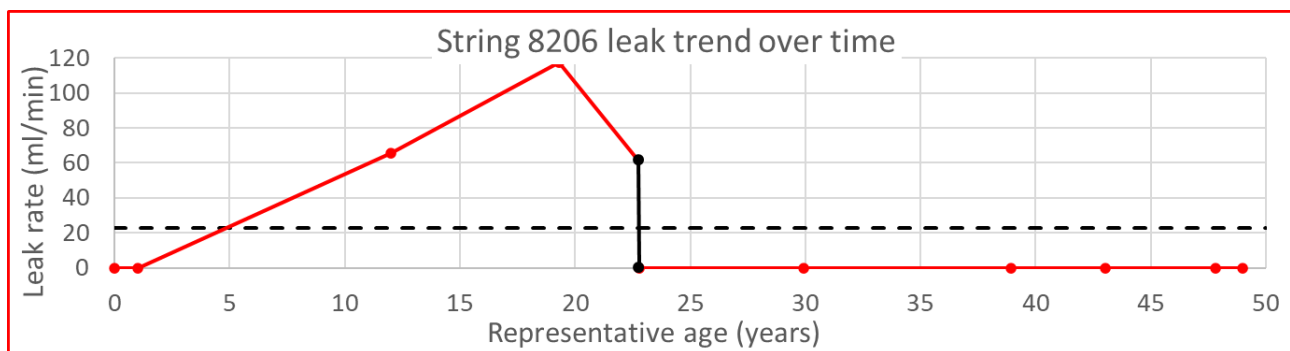


Figure 58: 8206 leak development over time

The leakage is from a valve stem, and is only present when the valve is open. This indicates that the 0% concentration in test 5 was likely due to experimental error, the most likely explanation that a valve was inadvertently opened while removing from the oven.

8.5 Non-hardening jointing compounds without support material (BS EN 751-2)

8.5.1 Overview of results

The results for the non-hardening compounds alone are presented in Figure 59. If a string had a joint retired, this also created a new string reference using the original string reference suffixed with the remaining joints. For example, 2202-3 became 2202-3ACDV after retirement of joint B. This is the same string, with the same remaining joints. The leak rate after retirement was taken as the new baseline leak rate for the new string.

String	Temp.	Baseline leak rate (ml/min)	Final leak rate (ml/min)	Increase in leak rate (ml/min)	Pass/Fail (and calculated leak rate from joints)	Ratio to failure rate
2201	20 °C	0.0	24.7	24.7	Fail D: 24.5 ml/min (0.0015 m ³ /hr)	Joint D (increase): 1.1
6201-1	60 °C	0.0	0.2	0.2	Pass	Total (increase): 0.0
8201	80 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
2202-3	20 °C	0.0	135.3	135.3	Fail B: 111 ml/min (0.0067 m ³ /hr)	Total (increase): 5.9 Joint B (increase): 4.8
2202-3ACDV	20 °C	24.7	12.8	-11.9	Pass	Total (increase): 0.0 Total (final): 0.6
6202-1	60 °C	0.0	406.0	406.0	Fail B: 406 ml/min (0.024 m ³ /hr)	Joint B (increase): 17.7
6202-1ACDV	60 °C	0.2	71.8	71.6	Fail A: 71.5 ml/min (0.0043 m ³ /hr)	Joint A (increase): 3.1
8202	80 °C	0.1	0.1 (92.6)	0.0	Fail Peak of 92.6 ml/min (0.0056 m ³ /hr) during testing	Total (increase to peak): 4.0
2203	20 °C	0.0	34.4	34.4	Fail D: 29.4 ml/min (0.0018 m ³ /hr)	Total (increase): 1.5 Joint D (increase): 1.3
6203-1	60 °C	0.0	33.6	33.6	Fail D: 33.4 ml/min (0.0020 m ³ /hr)	Total (increase): 1.5 Joint D (increase): 1.5
8203	80 °C	0.0	33.9	33.9	Fail Suspected C: 33.9 ml/min (0.0020 m ³ /hr)	Total (increase): 1.5 Suspected joint C (increase): 1.5
2204	20 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
6204	60 °C	0.0	0.2	0.2	Pass	Total (increase): 0.0
8204	80 °C	2.0	0.7	-1.3	Pass	Total (increase): 0.0
2207	20 °C	0.1	0.1	0.0	Pass	Total (increase): 0.0
6207	60 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
8207	80 °C	0.0	0.2	0.2	Pass	Total (increase): 0.0
2208	20 °C	0.0	0.2	0.2	Pass	Total (increase): 0.0
6208	80 °C	0.0	0.0	0.0	Pass	Total (increase): 0.0
8208	80 °C	0.1	1.2	1.1	Pass	Total (increase): 0.0
2210	20 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
6210	60 °C	0.0	3.7	3.7	Pass	Total (increase): 0.2
8210	80 °C	0.1	14.8	14.7	Pass	Total (increase): 0.6
2212	20 °C	0.0	0.2	0.2	Pass	Total (increase): 0.0
6212-1	60 °C	0.0	226.5	226.5	Fail C: 226 ml/min (0.014 m ³ /hr)	Total (increase): 9.8 Joint C (increase): 9.8

6212-1ABDV	60 °C	0.1	330.0	329.9	Fail A: 165 ml/min (0.0099 m ³ /hr) B: 165 ml/min (0.0099 m ³ /hr)	Total (increase): 14.3 Joint A (increase): 7.2 Joint B (increase): 7.2
8212	80 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
2214	20 °C	0.1	0.0	-0.1	Pass	Total (increase): 0.0
6214	60 °C	0.0	0.3	0.3	Pass	Total (increase): 0.0
8214	80 °C	0.0	23.5	23.5	Fail All joints: 23.5 ml/min (0.0014 m ³ /hr)	Total (increase): 1.0
2215	20 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
6215	60 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
8215	80 °C	0.2	0.2	0.0	Pass	Total (increase): 0.0

Figure 59: Final leak rates for non-hardening jointing compounds

There are several strings with leak rates above the 23 ml/min failure threshold. Some of these are only just above, and would pass if the leak was done using air (marked as marginal). The highest leak rate observed was 406 ml/min (0.024 m³/hr). The following sections discuss the sealants where leaks arose.

8.5.2 Hydrogen concentration prior to leak test

The hydrogen concentration recorded immediately prior to each scheduled leak test is given in Figure 60. It is clear that several of the strings did not retain high levels of hydrogen between leak tests. These are discussed individually with the discussion of the leaking strings in section 8.5.3. An isolated value of zero is likely to be due to experimental error. This can be seen with test 7 of 6201-1, which was due to the inadvertent opening of a valve. Note that the hydrogen concentration was returned to 100% after each leak test, and in the case of the strings with the largest leaks, was re-purged to hydrogen regularly between tests.

String ref	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
2201	88%	67%	81%	86%	86%	60%	63%	66%	76%
6201-1	87%	70%	79%	76%	78%	61%	0%	60%	60%
8201	95%	77%	75%	67%	77%	66%	73%	74%	78%
2202-3	89%	74%	88%	90%	69%				
2202-3ACDV						71%	84%	76%	76%
6202-1	72%	47%	77%	61%					
6202-1ACDV					70%	62%	56%	62%	64%
8202	91%	59%	70%	63%	34%	73%	55%	59%	73%
2203	65%	68%	35%	81%	14%	4%	3%	3%	12%
6203-1	44%	22%	56%	34%	1%	1%	0%	1%	0%
8203	1%	15%	3%	4%	1%	0%	1%	1%	5%
2204	89%	87%	80%	90%	81%	79%	80%	81%	82%
6204	81%	75%	75%	71%	73%	71%	70%	71%	74%
8204	59%	70%	63%	56%	53%	50%	55%	57%	56%
2207	86%	84%	70%	89%	76%	72%	82%	84%	84%
6207	95%	95%	81%	72%	79%	75%	73%	78%	79%
8207	71%	61%	34%	37%	26%	5%	16%	25%	38%
2208	92%	94%	91%	92%	93%	82%	82%	82%	86%
6208	94%	92%	83%	84%	82%	80%	78%	78%	81%
8208	64%	63%	63%	53%	53%	51%	50%	55%	39%
2210	92%	93%	89%	91%	92%	81%	81%	83%	85%
6210	77%	75%	76%	76%	67%	61%	59%	59%	63%
8210	69%	65%	2%	16%	0%	0%	3%	5%	14%
2212	94%	95%	81%	94%	87%	81%	83%	85%	82%
6212-1	73%	65%	75%	66%					
6212-1ABDV						59%	56%	6%	12%
8212	93%	2%	61%	57%	74%	72%	77%	77%	78%
2214	89%	91%	75%	89%	73%	74%	75%	76%	79%
6214	44%	52%	69%	43%	39%	7%	5%	23%	11%
8214	13%	15%	3%	3%	0%	0%	0%	0%	1%
2215	90%	89%	80%	92%	81%	80%	83%	83%	82%
6215	89%	88%	83%	82%	79%	79%	81%	81%	81%
8215	91%	83%	71%	72%	68%	68%	76%	76%	80%

Figure 60: Hydrogen retention for BS EN 751-2 – Non-hardening jointing compounds without support material

8.5.3 Analysis of leaking strings

The following sections provide a detailed review of each of the sealants that showed leakage.

Sealant 201

2201 leaked 24.7 ml/min (0.00148 m³/hr) at the final test. This string was used to demonstrate the relative leak rates of air, methane and hydrogen at a leak size around the failure threshold (see chapter 6.1 for details). The leak rate in air was 8.4 ml/min (0.0005 m³/hr). The leakage had been identified as coming from joint D. The day after measuring the relative leak rates, joint D was retired, to determine how much of the leakage was from D. At this point the total leakage had reduced to 19.5 ml/min. Retiring joint D reduced the leakage to 0.2 ml/min. The leak trend of 2201 is shown in Figure 61. The hydrogen concentration retained in 2201 was reasonably high.

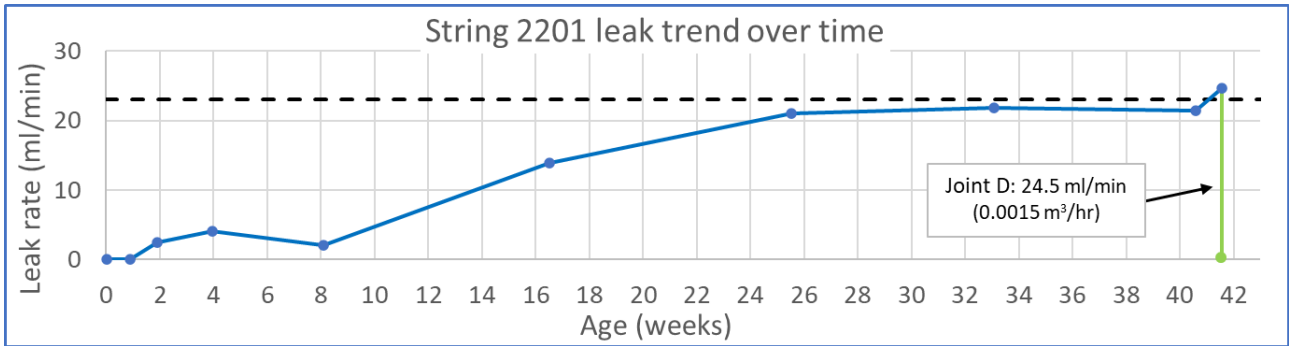


Figure 61: 2201 leak development over time

6201-1 had to be remade after week 1 tests. This followed an increase in leak rate from 0.0 ml/min to 25.0 ml/min (0.0015 m³/hr) on the original string 6201. 6201-1 had developed a leak rate of 25.2 ml/min at test 5 (aged 7.7 years), but this was identified as being from valve 1, and was removed entirely when the valve was changed. 6201-1 is the only string in the test programme that developed a leak after one week, that did not then develop a leak in the remaining test schedule after being re-assembled with a new reference.

The concentration in both these strings remained high, except a single result for 6201-1 in test 7. The experimental notes for this test indicate that the valve was open when the string was prepared for concentration testing. It was unclear how long the valve had been open for.

The results for the 60°C and 80°C strings for sealant 201 showed no sign of leakage. The leak trend graphs for both temperatures are provided in Figure 62.

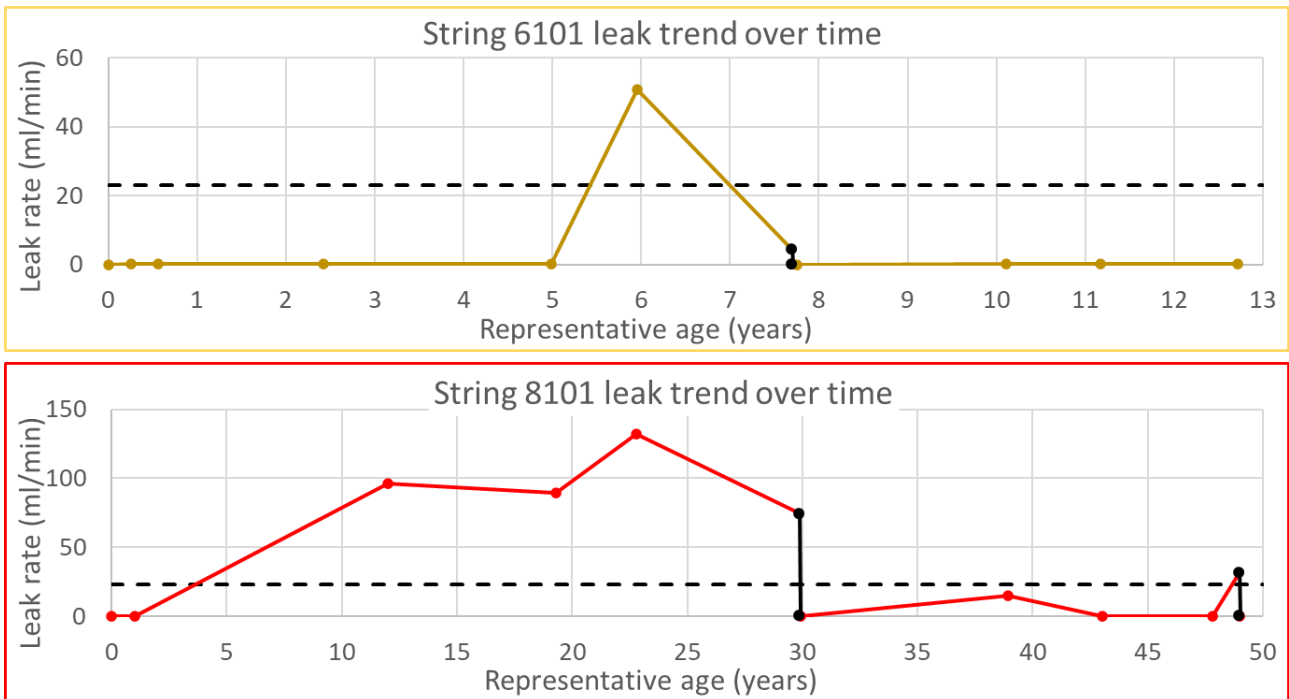


Figure 62: 6201-1 and 8201 leak development over time

While both strings showed valve leakage, there was none from the test joints. Overall, these results indicate that hydrogen incompatibility is unlikely, as only one of twelve joints failed, it was marginally higher than the failure rate, and it was the 20°C string that developed a leak. If the leak were due to chemical reactions with hydrogen, this would also be seen at the higher temperatures.

Sealant 202

Both 2202 and 6202 developed leaks after the first week. 2202 started with zero baseline leakage, but developed a leak of 388.8 ml/min (0.023 m³/hr) by the time of test 1. It was remade and included in batch 3 as 2202-3, with zero baseline leakage. Note that several attempts were made with this string, using a variety of

fittings, to achieve a leak free baseline. 6202 increased from zero baseline leakage to 155.9 ml/min (0.0094 m³/hr) after one week (aged 91.9 days). This string was removed, and replaced with 6202-1.

2202-3 developed a large leak of 135.3 (0.0081 m³/hr), and joint B was retired after 151 days (both real and representative age). Leakage was reduced to 24.7 ml/min (0.0015 m³/hr), indicating a final leak rate from joint B of 110.6 ml/min (0.0066 m³/hr). The string with B retired continued in the test programme named 2202-3ACDV, with 24.7 ml/min as the baseline leak rate and the final leak rate was 12.8 ml/min. During test 6, just after 26 weeks, the leakage was noted as being from joints A and B. This datapoint is therefore potentially invalid, as it includes leakage from the already retired joint B. for the following test in week 32, joint B was re-sealed. During the final leak test, joint B was re-retired twice, with leakage reducing from 16.6 ml/min to the 12.8 ml/min final leak rate. This indicates that the remaining leakage was not from B. Applying leak detection fluid demonstrated clearly that the leak was coming from joint A. No leakage was detected from joints C and D, or the valves. The leak trend of 2202-3 is shown in Figure 63.

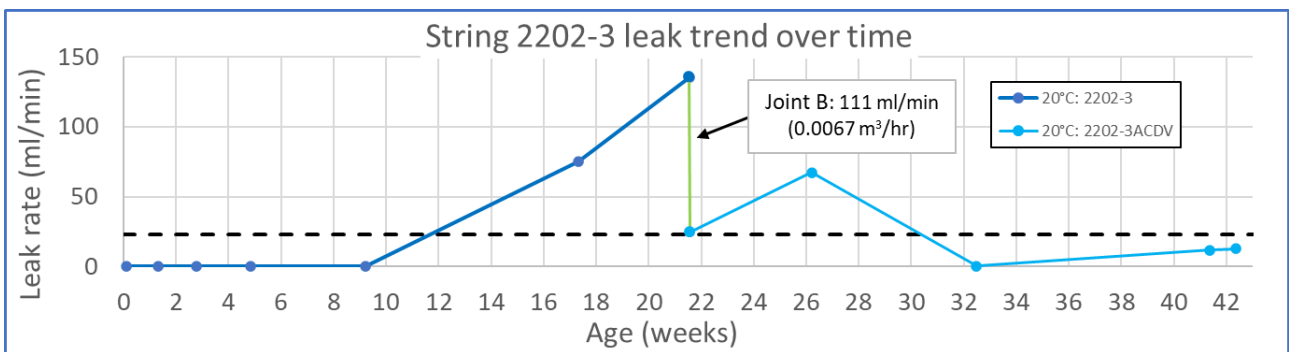


Figure 63: 2202-3 leak development over time

6202-1 developed a leak of 406.0 ml/min (0.024 m³/hr). This was the largest test joint leak found during the test programme. Joint B was retired when the representative age was approximately 5 years old, accounting for all of that leak. The remaining string 6202-1ACDV slowly increased in leak rate, eventually leaking at 71.8 ml/min (0.0043 m³/hr). During final test retirement joint B was re-retired, but with no decrease in leak rate. Leakage was identified as being from joint A, and when this was retired the leak rate dropped to 0.3 ml/min. The leak trend of 6202-1 is shown in Figure 64.

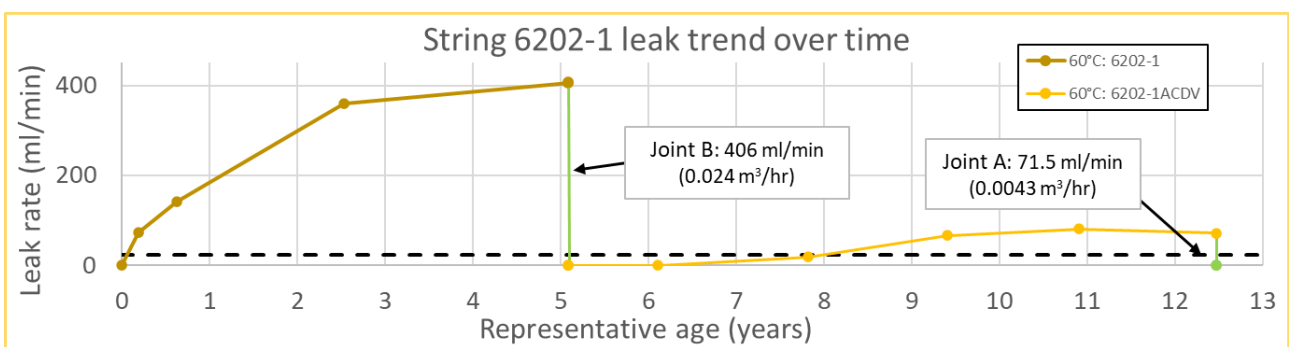


Figure 64: 6202-1 leak development over time

The leak trend of 8202 is shown in Figure 65.

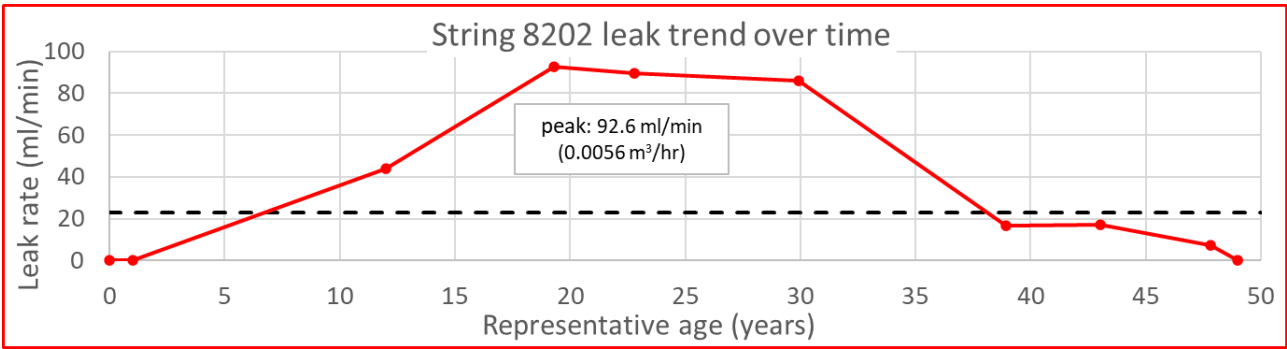


Figure 65: 8202 leak development over time

While 8202 demonstrated no leakage at the final test, the leak rate during the test programme peaked at 92.6 ml/min (0.0056 m³/hr). This leakage is believed to be from valves and from test joints. Notes taken during test 4 (23 years) indicate that leakage was from valve 2 and joint C or D. Similar notes for test 5 (30 years) indicate leakage from mostly C and D, with some from A or B. No change was made for test 6, when the leakage decreased to 17.5 l/min (0.0011 m³/hr). In test 7 the initial leak rate for 8202 was 55 ml/min, with leakage detected from the stem of valve 1. The string was rotated 180° to use valve 2 as the input. The leak rate increased to 130 ml/min. Both valves were removed and replaced. When tested again in the normal orientation (valve 1 upstream), the leak rate was initially 27 ml/min, which dropped to 18 ml/min over the course of around three minutes. Note that this was still decreasing at this point. The raw pressure and flow trace for this final attempt is provided in Figure 66.

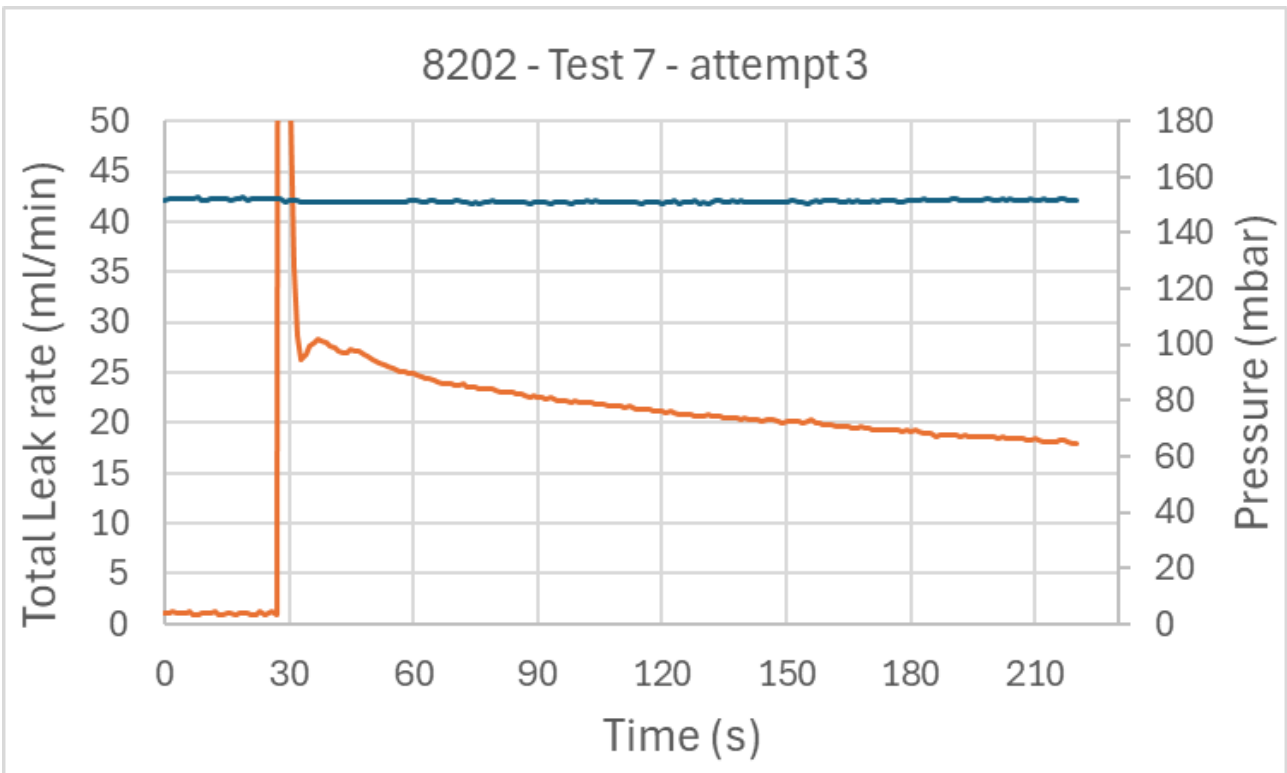


Figure 66: 78202-3 - String 8202 Test 7, attempt 3

This is an unusually slow decay in flow rate, with flow rate generally stabilising within approximately 10 seconds. While the final increase in leak rate for 8202 was minimal, at least part of the leakage spike during the test was likely to be from test joints.

Sealant 203

All three 203 strings had no baseline leakage, but all developed leaks marginally above the failure threshold. The leak trend of 2203 is shown in Figure 67.

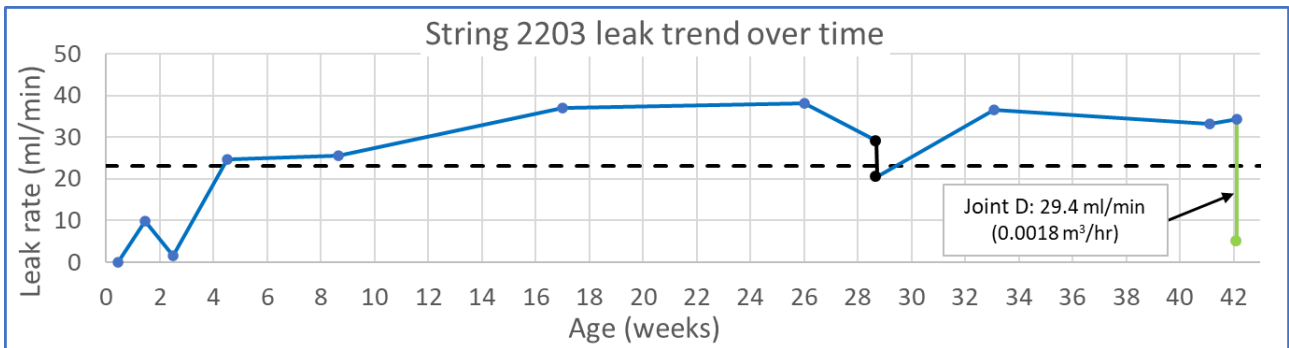


Figure 67: 2203 leak development over time

Valve 1 was replaced between tests 6 and 7, when the string was approaching 0.6 years old. Leakage was reduced from 29 ml/min (0.0017 m³/hr) to 20.5 ml/min (0.0012 m³/hr). Note that this step was done outside of the scheduled testing, and the leak rate measured at that time was slightly lower than the leakage previously observed.

In the final leak test, the leakage was identified as being from joint D. Retiring this reduced the leak rate from 34.4 to 5.0 ml/min, indicating that joint D had a 29.4 ml/min (0.0018 m³/hr) leak. The leak rate development for string 6203-1 is given in Figure 68.

String 6203-1 was part of Batch 3, as the original string leaked at 50 ml/min (0.003 m³/hr) at the first test (increased from a baseline leak rate of zero). This had been in the oven for one week, but was aged approximately 115 days. The replacement string also began to leak by a similar amount after a week. The leak rate spiked to 56.1 ml/min, before stabilising to approximately 26 ml/min (0.0016 m³/hr). The results for string 6203-1 are shown in Figure 68.

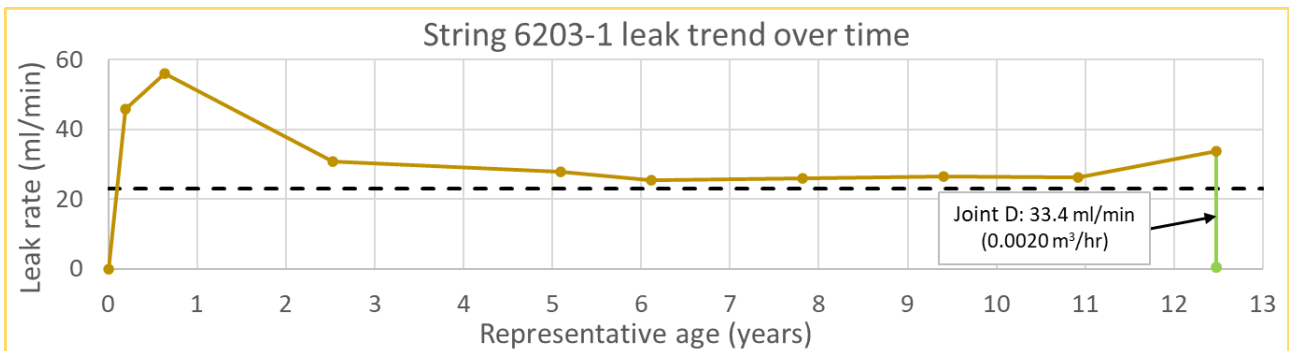


Figure 68: 6203-1 leak development over time

The reason for this leak rate spike early in the test programme is not known. Identification of leak location was not carried out in these early tests, and no experimental notes regarding this were made at the time. The valves were not changed or re-sealed between tests 2 and 3, but variability in valve leak rate from test to test has been observed in a number of other strings. No leakage was identified as being from the valves during the test programme.

The final leak rate was 33.6 ml/min (0.0020 m³/hr). This was identified as being from joint D, and when retired this reduced the leak rate to 0.2ml/min.

The results for string 8203 are shown in Figure 69.

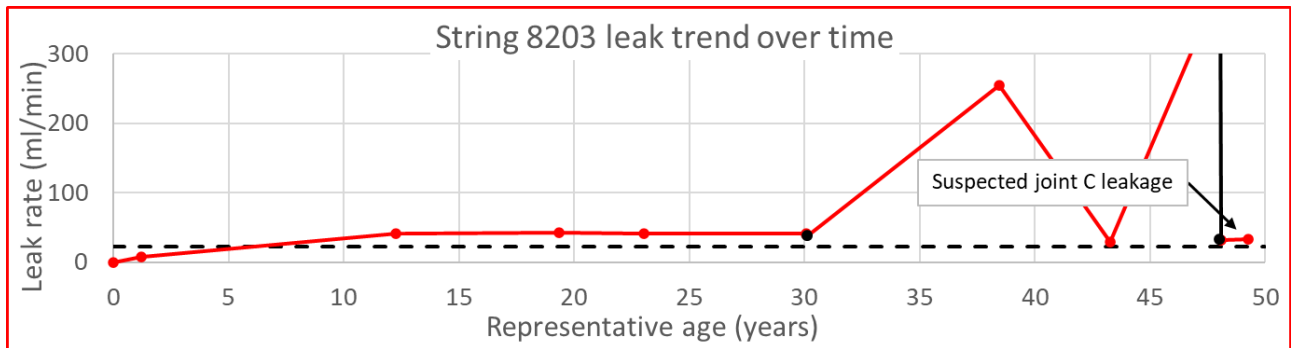


Figure 69: 8203 leak development over time

Leakage from 8203 stabilised at around 30 ml/min (0.0018 m³/hr). Valve 1 was replaced for test 5, but the leak rate was not significantly reduced. The spike to 254 ml/min (0.015 m³/hr) in test 6 was a temporary feature, which reduced again to a similar level as previously. This spike is typical of intermittent valve stem leakage (with the valve open), but this could not be confirmed during the test. Valve 1 was replaced again between tests 7 and 8, at an age of approximately 48 years. While the officially recorded leak rate at test 7 was 29.1 ml/min, the leak rate at the start of the valve replacement was 400 ml/min (0.024 m³/hr). Changing the valve reduced this to 32.8 ml/min (0.0020 m³/hr).

Stroking the valve was believed to cause intermittent stem leaks with the valve open, with the leak rate from the test joints was averaging approximately 30 ml/min.

During the final test, leakage was noted as emanating from joint C. An attempt to undo and retire joint C was made, but the sealant had cured very hard, and the lugs on the brass barrel were rounded while trying to loosen. This string is shown in Figure 70. No further attempts to disassemble 8203 have been made.



Figure 70: 8203 non-hardening sealant that cured hard

It should be noted that while the manufacturer states that sealant 203 is non-setting and can be used as a thread sealant, it is unlikely to be used as such often and would more commonly be used as a flange sealant.

Sealant 212

String 2212 sealed well at the start, and did not develop any leaks during the test programme. The leakage of 2212 is shown in Figure 71.

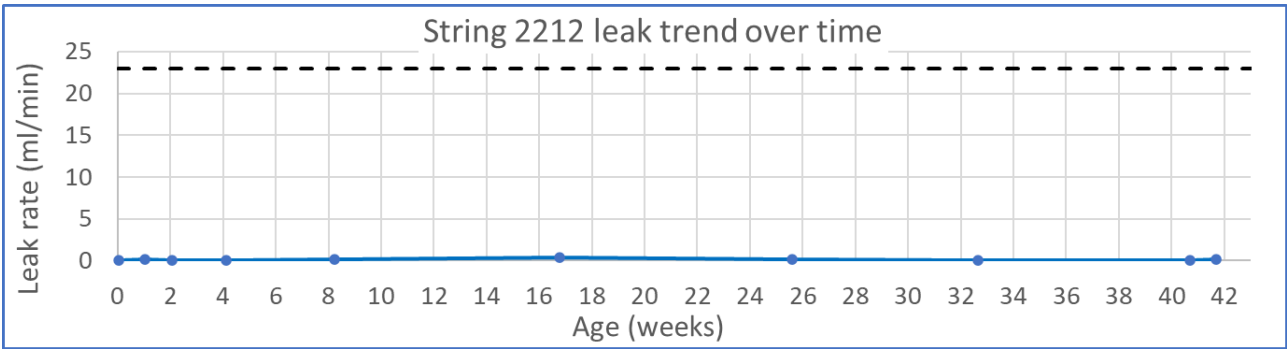


Figure 71: 2212 leak development over time

6212 developed a leak of 98.8 ml/min (0.0059 m³/hr) after week 1 (aged 112 days). This was therefore removed and the string reassembled as 6212-1. 6212-1 also developed leakage almost immediately, increasing from a baseline of 0.0 ml/min to 115.9 when aged 70 days.

By test 4, 6212-1 was leaking 226.5 ml/min (0.014 m³/hr). The leakage was identified as being from joint C, so this was retired, initially reducing the leakage of the string to 0.1 ml/min. The string was renamed 6212-1ABDV.

Joint C did not remain sealed however, with increased leakage seen in subsequent tests. A further attempt to retire joint C was made between tests 6 and 7, temporarily reducing the leakage to 29.2 ml/min (0.0018 m³/hr). By the next test however this had increased to 338.7 ml/min (0.020 m³/hr). At this stage it had been accepted that the retirement method was not successful, so the retirement process was not repeated.

During the final test, joint C was disassembled and remade using a 1-inch bonded washer. This provided a temporary seal, enabling the leak rate for the remainder of string 6212-1ABDV to be measured.

The final leak rate of string 6212-1ABDV (with joint C sealed) was 330 ml/min (0.020 m³/hr). Joint A was retired, and upon testing the leakage had reduced to 164.7 ml/min (0.0099 m³/hr). Although switching measurement systems introduces some error, the approximate leakage attributable to joint A was 165.3 ml/min. Retiring joint B then reduced the leakage to 0.0 ml/min, showing that joint D (plus the valves) were fully sealed.

The development of leakage in string 6212-1 and subsequently 6212-1ABDV is shown in Figure 72.

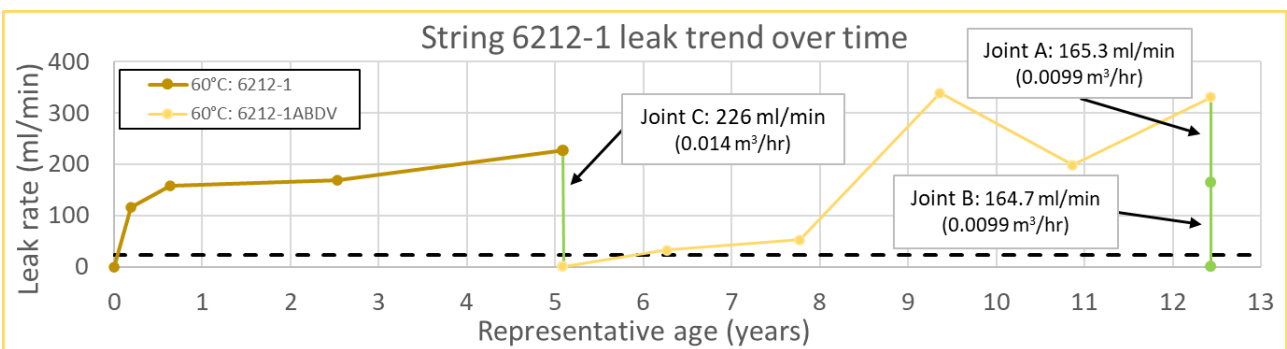


Figure 72: 6212-1 leak development over time

8212 leaked at a rate of 89.9 ml/min (0.0054 m³/hr) at test 2, when it was the equivalent of 12.3 years aged. This leakage reduced to 2.1 and 2.4 ml/min in the subsequent tests, before reducing further. The valves were not changed, and the reason for the change is not known. The hydrogen concentration recorded at these leak tests was low, indicating it was more likely to be joint leaks than a valve stem leak – a valve stem leak is generally only present when the valve is open, so would not manifest while the string is in the oven between tests. The results for string 8212 are shown in Figure 73, including the concentrations recorded.

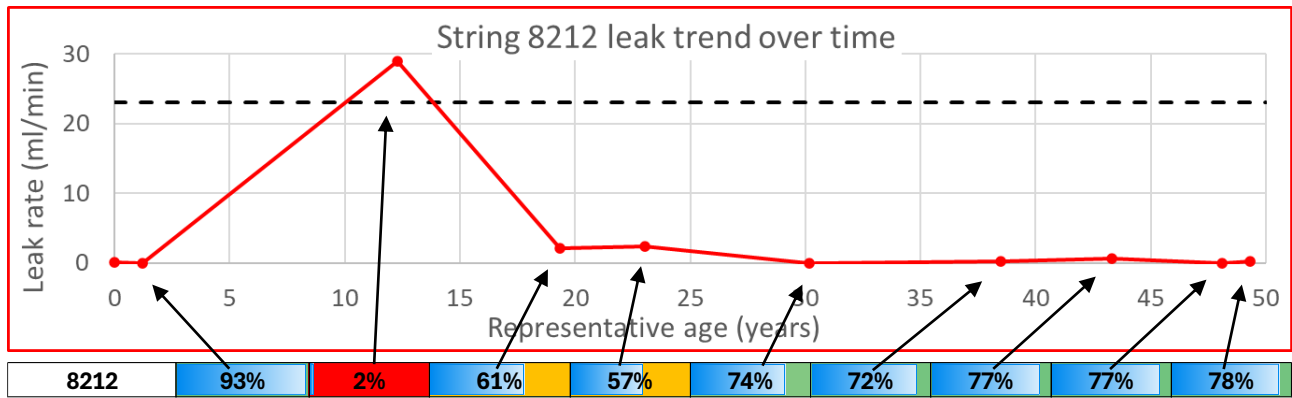


Figure 73: 8212 leak development over time

Sealant 214

String 8214 reached just over the threshold with a final leak rate of 23.5 ml/min (0.0014 m³/hr). The graph showing the leakage over time is given in Figure 74.

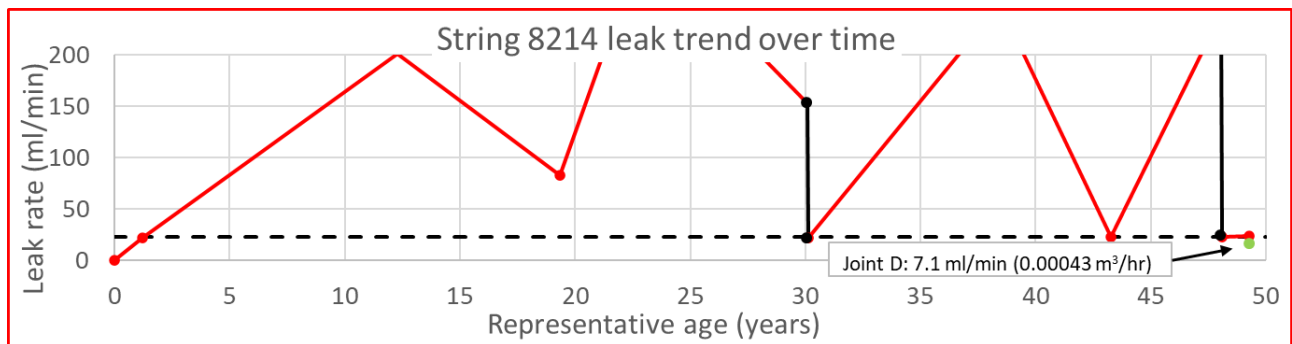


Figure 74: 8214 leak development over time

The high leak rates measured were attributed to leakage from the stem of Valve 1, which was replaced twice. The string still leaked however. During the final test (after also testing with air and methane), joint D was retired, reducing the leakage from 23.5 ml/min to 16.4 ml/min (0.00098 m³/hr). This attributes 7.1 ml/min (0.00043 m³/hr) of leakage to joint D. As the leakage was under the threshold, a further breakdown was not carried out. Joint D appeared to be the source of the largest leak, with the rest expected to be fairly evenly distributed.

8.6 Unsintered PTFE tapes and cords (BS EN 751-3)

8.6.1 Overview of results

The final leak rate for each string with PTFE tape is given in Figure 75. Where strings had joints retired, the final leak rate at that point is shown for the originally named string, then the modified string is shown below with the ongoing result for the remaining joints. The leak rate inferred for each joint during the retirement process is shown on the test cards but not included here.

String	Temp.	Baseline leak rate (ml/min)	Final leak rate (ml/min)	Increase in leak rate (ml/min)	Pass/Fail (and calculated leak rate from joints)	Ratio to failure rate
2205	20 °C	0.1	0.1	0.0	Pass	Total (increase): 0.0
6205	60 °C	9.1	11.3	2.2	Pass	Total (increase): 0.1
8205	80 °C	0.0	0.8	0.8	Pass	Total (increase): 0.0
2209	20 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0

6209	60 °C	6.7	19.2	12.5	Pass	Total (increase): 0.5
8209	80 °C	0.0	12.6	12.6	Pass	Total (increase): 0.5
2301	20 °C	0.4	0.7	0.3	Pass	Total (increase): 0.0
6301	60 °C	11.9	16.0	4.1	Pass	Total (increase): 0.2
8301	80 °C	13.2	10.8	-2.4	Pass	Total (increase): 0.0
2302	20 °C	0.1	0.2	0.1	Pass	Total (increase): 0.0
6302	60 °C	0.0	-0.1	-0.1	Pass	Total (increase): 0.0
8302	80 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
2303	20 °C	43.6	39.9	-3.7	Never sealed A: 26.9 ml/min (0.0016 m ³ /hr) B: 12.7 ml/min (0.00076 m ³ /hr) C: 5.1 ml/min (0.00031 m ³ /hr) D: 3.0 ml/min (0.00018 m ³ /hr)	Total (final): 1.7 Joint A (final): 1.2 Joint B (final): 0.6 Joint C (final): 0.2 Joint D (final): 0.1
6303	60 °C	348.6	270.0	-78.6	Never sealed A: 20.0 ml/min B: 269 ml/min	Total (final): 11.7 Joint A (final): 0.9 Joint B (final): 11.7
6303CDV	60 °C	0.7	0.7	0.0	Pass	Total (final): 0.0
8303	80 °C	288.7	240.0	-48.7	Never sealed A: 34.8 ml/min B: 19.0 ml/min C: 163 ml/min	Total (final): 10.4 Joint A (final): 1.5 Joint B (final): 0.8 Joint C (final): 7.1
8303DV	80 °C	3.1	10.8	7.7	Pass	Joint D (final): 0.5
2304	20 °C	0.1	0.0	-0.1	Pass	Total (increase): 0.0
6304	60 °C	0.0	0.1	0.1	Pass	Total (increase): 0.0
8304	80 °C	0.3	0.1	-0.2	Pass	Total (increase): 0.0

Figure 75: Final leak rates for PTFE Tapes

The results show that there is no marked increase in leak rate over time. The thin PTFE Tape strings (303) did not seal well from the start, and all three showed a decrease in leak rate.

8.6.2 Analysis of leaking strings

Assembling the strings with zero leakage joints was challenging with the PTFE tape alone, but no issues were noted when non-hardening jointing compound was also used. Baseline leakage was measured after assembly, with a wait period for those with compound. All the strings with only PTFE tape leaked from when they were first assembled.

205 & 209 (PTFE cord with no compound)

Both types of PTFE cord with no compound had one string with a small amount of leakage from the start. This was 9.1 ml/min for 6205, and 6.7 ml/min for 6209. The 205 strings did not increase significantly, but two

209 strings showed a moderate increase in leak rate over the test program. This was an increase of 12.5 in string 6209 and 12.6 ml/min in 8209.

No leakage was observed from string 2209. This is shown in Figure 78.

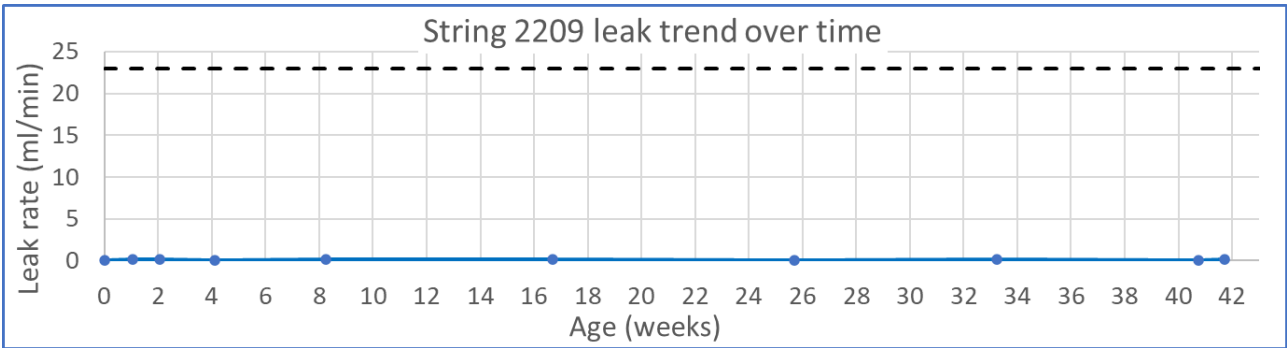


Figure 76: 2209 leak development over time

The leak results of 6209 in Figure 77 show that the leak rate increased early in the test programme, but stabilised below the failure threshold. It is unclear whether this increase was due to the presence of hydrogen.

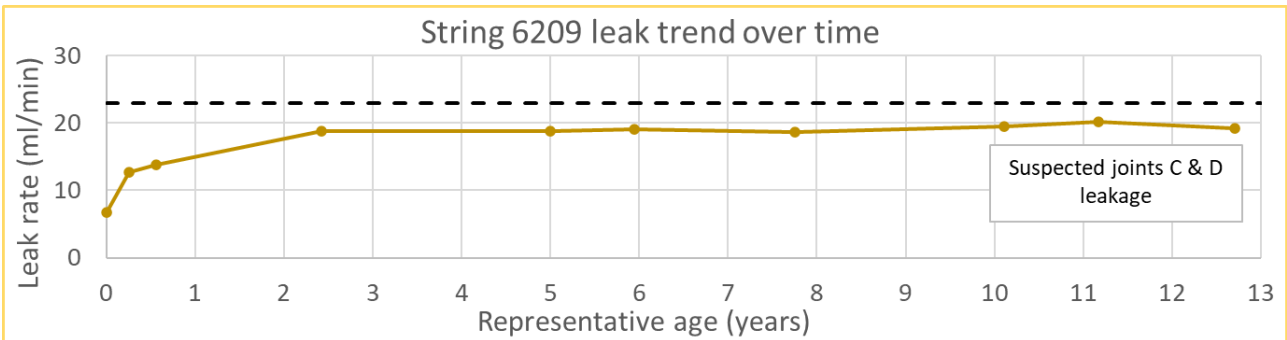


Figure 77: 6209 leak development over time

The leak results of 8209 in Figure 78 show a similar pattern to 6209, but with additional valve leakage. The reduction in leak rate to a consistent value when the valves were replaced is a strong indication that the leak rate is from a test joint.

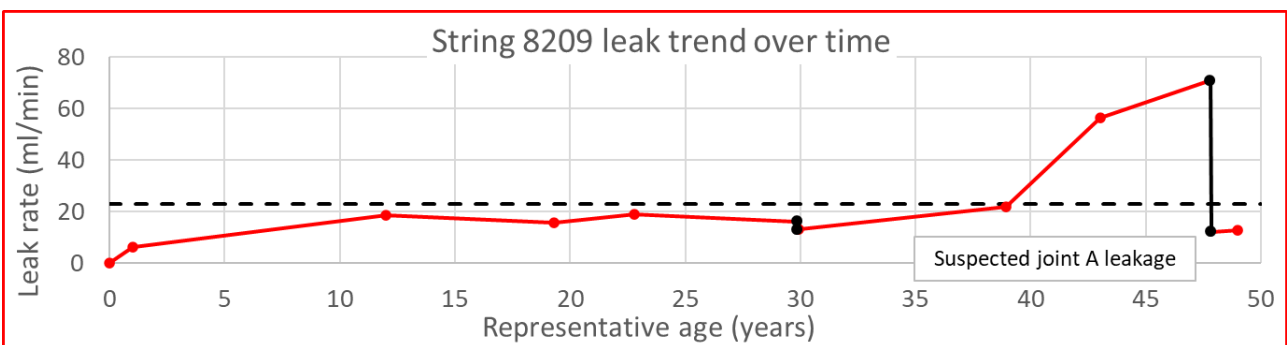


Figure 78: 8209 leak development over time

All joints with PTFE cord alone were below the failure rate, but did not seal as well as when used in conjunction with compound.

301 Thick (yellow) PTFE tape

The 301 strings are those with only thick (yellow) PTFE tape. This is currently allowed to be used on threaded fittings for gas installations. All three 301 strings showed some leakage from when they were first assembled. The starting leak rate was below the fail rate, but it was significantly higher than the anaerobic sealants or

combinations. 2301 was only 0.4 ml/min (notable, but below the calibrated minimum flow reading), but 6301 and 831 leaked at 11.4 ml/min and 13.2 ml/min respectively.

String 2301 did not develop any leakage. The leak rate trend for 2301 is shown in Figure 79.

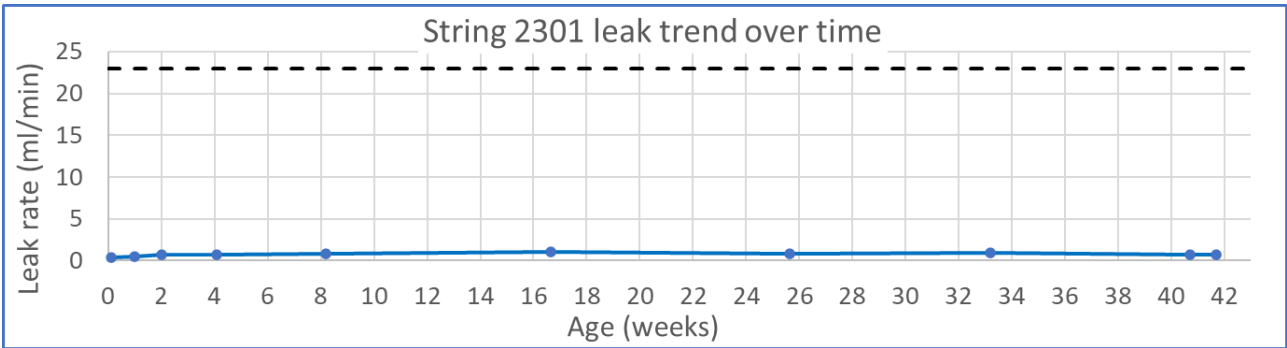


Figure 79: 2301 leak development over time

The development of leakage in string 6301 is shown in Figure 80. Although the leak rate is moderate throughout, it does not get significantly worse.

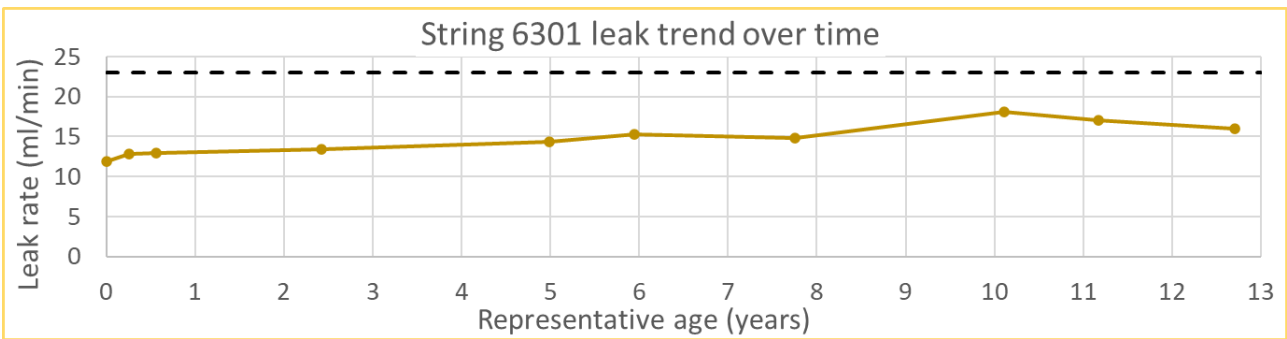


Figure 80: 6301 leak development over time

8301 is shown in Figure 81. The pattern is similar to 6301, but with the addition of suspected valve leaks.

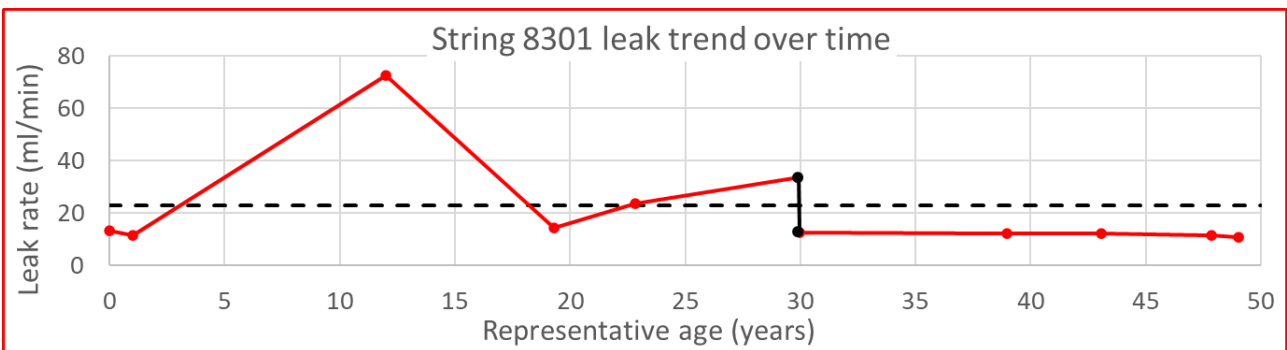


Figure 81: 8301 leak development over time

303 Thin (white) PTFE tape

It should be noted that thin (white) PTFE tape is not recommended for use on gas installations, but there is an expectation that it has been used in the past, is likely to still be used by some fitters.

For baseline leakage, 2303 leaked at 43 ml/min (0.0026 m³/hr), 6303 at 348.6 ml/min (0.021 m³/hr) and 8303 at 288.7 ml/min (0.017 m³/hr). All three 303 strings (thin PTFE tape) failed the overall test before hydrogen was introduced. Several attempts were made to reassemble these with no leakage, but eventually they were included with leakage. The hydrogen concentration was expected to remain high even if the pressure dropped quickly.

Following the findings of the hydrogen retention tests however, it was evident that this was not the case. External sealing was not sufficiently retaining hydrogen in these strings, so the leakiest joints were retired in an attempt to obtain results for the remaining joints.

The leak results for 2303 is shown in Figure 82, 6303 in Figure 83, and 8303 in Figure 84.

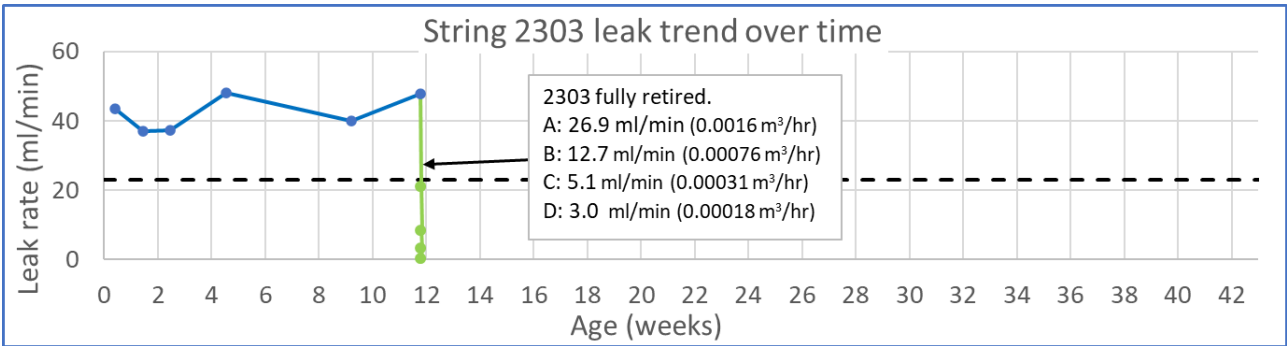


Figure 82: 2303 leak development over time

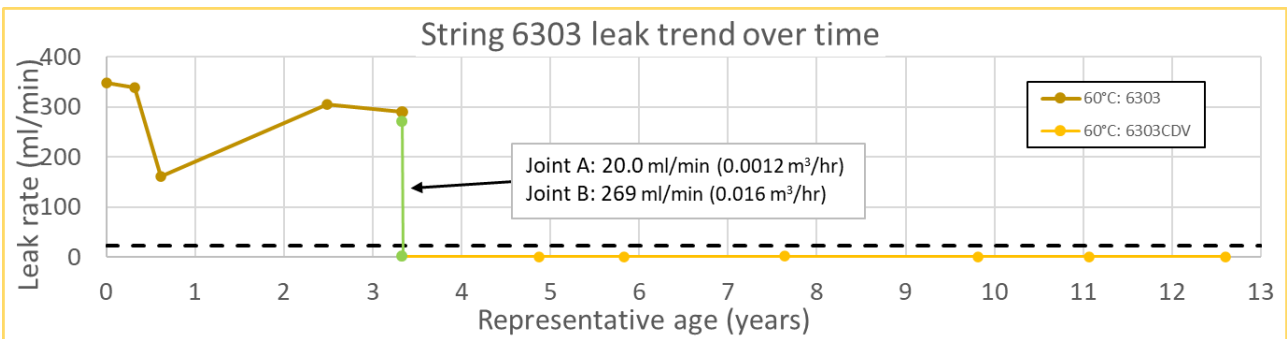


Figure 83: 6303 leak development over time

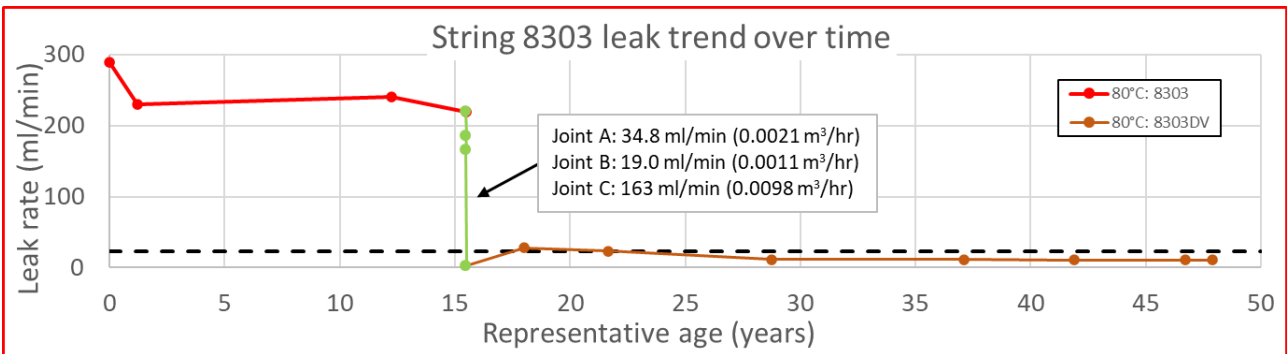


Figure 84: 8303 leak development over time

Although the initial leak rates are high, there is no indication that the leak rate increased, or that there is any incompatibility with hydrogen.

8.6.3 Hydrogen concentration prior to leak test

The hydrogen concentration recorded immediately prior to each scheduled leak test is given in Figure 85. It is clear that the 301 and 303 strings were not retaining hydrogen. This resulted in retirements for these strings, as insufficient hydrogen concentration was being retained to ensure the compatibility test was valid. External wrapping had been used in an attempt to retain more hydrogen, but this was not sufficient for these strings. It is notable that the concentration retained in 6303 and 8303 remained low after the retirements. The concentration was low in 2301 as well, although the leak rate was very low at approximately 0.4 ml/min. This may indicate that PTFE tape allows a faster rate of air ingress than compounds, for an equivalent leak rate.

String ref	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9
2205	95%	94%	89%	92%	90%	79%	80%	80%	86%
6205	71%	68%	68%	59%	49%	23%	28%	42%	38%
8205	92%	75%	77%	76%	78%	75%	77%	79%	82%
2209	97%	92%	88%	91%	89%	69%	71%	72%	69%
6209	34%	73%	56%	55%	47%	6%	4%	14%	10%
8209	27%	63%	39%	36%	15%	2%	10%	9%	38%
2301	6%	15%	19%	45%	16%	3%	3%	4%	13%
6301	3%	6%	9%	7%	9%	1%	8%	1%	1%
8301	3%	5%	4%	3%	1%	0%	0%	0%	3%
2302	92%	94%	78%	93%	80%	82%	81%	80%	83%
6302	93%	92%	86%	84%	83%	80%	82%	84%	82%
8302	88%	81%	66%	70%	54%	39%	50%	52%	52%
2303	3%	15%	14%	35%					
6303	2%	14%	57%						
6303CDV				6%	6%	1%	10%	1%	1%
8303	3%	25%							
8303DV			3%	4%	1%	0%	1%	1%	6%
2304	93%	93%	79%	93%	82%	81%	83%	84%	83%
6304	90%	88%	71%	35%	50%	37%	30%	38%	36%
8304	91%	83%	68%	74%	71%	54%	59%	60%	50%

Figure 85: Hydrogen retention for BS EN 751-3 – Unsintered PTFE tapes

8.7 Summary of results

The majority of the strings passed, with 8 strings of 75 developing an increase in leak rate that failed, and a further three having an initial leakage that did not significantly increase. There were a total of 300 joints within the 75 strings. Of these, a total of individual 12 joints developed failure leak rates (including two that were on strings with a joint already retired).

None of the leaks measured were large leaks, the highest being 406 ml/min, or 0.024 m³/hr. As these are threaded joints, any leak path will be convoluted and mostly filled with a viscous paste.

A total of 37 valve replacements were carried out, with some leak tests also being carried out with the string in reverse to eliminate the leakage through the stem of valve 1, which was only present with the valve open. This represents a potential hazard, though anecdotal evidence from industry experts indicates that this type of valve are known to leak in such a way with Natural Gas in current usage.

It was notable that all of the strings that failed were part of the group with non-hardening compounds with no support material. The inclusion of support material such as PTFE cord or tape therefore appears generally to improve the reliability of non-hardening compounds.

8.7.1 Sealant groups that passed

No leakage was observed from the strings with anaerobic sealants. The results of these experiments indicate that the sealants tested are suitable for use with hydrogen in domestic and commercial installations.

All strings where support material and compound were used together passed the leak tests, with no leakage observed. This was with hemp, PTFE tape or PTFE cord as support, and with a range of compounds.

The strings with PTFE cord and no compound showed signs of leakage both initially and developed during the test programme, but none of them failed. This is unlikely to be due to degradation caused by exposure to hydrogen, but due to the lack of fluidity in the sealing substance.

Five of the ten non-hardening compounds without support material passed the leak tests. Of these, one did develop some leakage in two strings. Two of those that technically failed were only just above the failure rate.

All three strings with thin (white) PTFE tape (code 303) alone had a baseline leak rate above the failure rate, while the thick (yellow) PTFE tape alone (code 301) showed some leakage. None of these increased by a significant amount however. This implies that although PTFE tape alone may not be suitable for threaded gas fittings, these results do not show any evidence that PTFE tape was degraded by hydrogen.

8.7.2 Sealants that leaked

The nine strings that developed leaks beyond the failure threshold are all from the group of BS EN 751-2 non-hardening jointing compounds with no support material. These are:

- 2201
- 2202-3
- 6202-1
- 8202
- 2203
- 6203-1
- 8203
- 6212-1
- 8214

The highest leak rates were from 202, which failed with multiple joints and strings, and 212 which had multiple failures within a single string. All three 203 strings failed, but only marginally. While the cause for these failures is not known, an adverse chemical reaction with hydrogen remains a possibility.

The total string leak rate for strings 2201 and 8214 was only just above the failure rate. For 2201 this was entirely from one joint, but on 8214 it was shared between at least two joints. The leakage from individual joints on 8214 was therefore below the threshold and there is no clear indication that hydrogen is the cause for sealant deterioration. With 201, the leakage was only on the 20°C string, indicating that it is very unlikely that chemical reactions with hydrogen was the cause. Sealants 201 and 214 do not appear to have an adverse reaction to hydrogen.

Sealant 210 passed, however some leakage developed on both the 60°C and 80°C strings. The cause for this leakage is unknown.

It is notable that in general the strings that developed leakage after one week and were subsequently remade developed failing leak rates in the newly made string. 6201-1 is the only string that did not subsequently develop a leak, and passed overall. The propensity for the same strings to develop leaks at the second attempt may indicate an issue with the sealant or with the fittings. The fittings were changed for the remade strings, to reduce the likelihood that the fault is with the threads.

8203 clearly hardened over time. This is marketed as a non-hardening sealant, and one that should remain soft. It is possible that the leakage seen in all three 203 strings is a result of the sealant curing hard and drying. This would explain why there was an increase in leak rate but not to a high level. It is unclear if this hardening is a result of contact with hydrogen, a reaction with air, or another property of the sealant. Leakage began within a week of the joints being made, and did not then increase significantly. There was no significant difference in maximum leak rate between the temperatures. The product is rated as suitable for up to 250°C, so the difference in temperature should not be a significant factor. Discussions with the manufacturer at the end of the test programme highlighted that although the sealant is designed to be non-hardening, and is advertised as being suitable for thread sealing, this is not its primary purpose, and it is not compliant with BS EN 751-2.

8.7.3 Compound used with and without support material

There were three non-hardening jointing compounds tested both with and without support material. Two of these without support failed, while their equivalent with PTFE cord passed. These were sealants 211 with PTFE cord as support, and 212 without ; and 213 with, and 201 without PTFE cord. The third non-hardening compound used with (206) and without (207) PTFE cord as support developed no leakage.

This provides a strong indication that using PTFE cord as support increases the reliability of non-hardening jointing compounds, although some may not need it. Whether a non-hardening compound is improved by having PTFE cord is likely to be related to the viscosity. It is anticipated that sealants that remain highly viscous after prolonged periods are less likely to require support materials.

9 Conclusions and recommendations

None of the anaerobic sealants showed any evidence of deterioration or leakage. None of the non-hardening compounds with support material showed any sign of leakage. PTFE tape alone did not seal well, but did not deteriorate. When PTFE tape was used in conjunction with compound, no leakage was detected.

Of the nine non-hardening compounds tested without support materials such as PTFE tape or PTFE cord, four did not develop any leakage in any of the test pieces. Two more developed leakage in a single test piece at around the failure rate, but the indications were that this was not caused by chemical reactions with hydrogen.

The use of support with non-hardening compounds appears to significantly improve the reliability, particularly with less viscous sealants. Without further investigation it is unclear whether the cause of failures was hydrogen incompatibility, or sealant performance in general.

The leak rates measured in this study are small, with the largest observed being 406 ml/min, or 0.024 m³/hr. This is estimated to be just less than the leakage expected from a 0.025 mm² hole at 150 mbar.

9.1 Sealant suitability

During development of the list of sealants to test in this project, some of the sealant manufacturers noted that some products were approved for use with hydrogen. A review of the techniques used was not carried out during this study, but this highlighted the difference between comparative leak testing and testing for chemical compatibility. This test programme is concerned with chemical compatibility.

The list of sealants that passed are provided below (alphabetical order only, not related to test number).

9.1.1 BS EN 751-1

None of the anaerobic jointing compounds tested developed any leakage. These are:

- Cimberio C70-10
- Loctite 577
- Loxeal FR58-11
- Loxeal FR58-11 with Activator 11
- Rocol Rapidseal

9.1.2 BS EN 751-2

All non-hardening jointing compounds that were used with support material (hemp, PTFE cord and PTFE tape) passed, with no leakage.

The non-hardening jointing compounds used on their own that did not show any sign of deterioration due to contact with hydrogen are:

- Boss White
- Facot Silicon tape
- Fernox Hawk White
- Fernox Water Hawk
- Rocol Gasseal
- Stag Jointing Compound – Type B

Of these, four developed no leakage, while two developed leaks in a single string of approximately the stated failure rate. The leakage appears to be unrelated to hydrogen, so these sealants have been included in this list. A seventh non-hardening compound also developed multiple small leaks (below the failure threshold,

but measurable) over the test programme. It is unclear whether a reaction with hydrogen was the cause, so this sealant is not listed.

Three non-hardening sealants developed leakage in a number of joints and/or test strings. This could be related to hydrogen, though further investigation would be required to determine whether this is the case.

Two of the non-hardening compounds that developed leakage when used alone did not when used with PTFE cord as support. Whether or not hydrogen was the cause for the leakage developing, the use of support material appeared to mitigate that risk.

9.1.3 BS EN 751-3

Thick yellow PTFE tape in conjunction with a non-hardening jointing compound passed with no leakage detected. Thin, white, PTFE tape does not comply with the standards, although when used in conjunction with compound, it also showed no leakage. Both types of PTFE tape, and PTFE cord alone (without compound) leaked from when they were first assembled, but did not appear to deteriorate.

9.2 Use of support material with non-hardening compounds

Support material refers to an additional component added to the joint to form a solid blockage, or matrix, that bridges larger gaps in the joint. Traditionally this would have been hemp but is now more likely to be PTFE cord. The non-hardening compound or paste fills smaller gaps between the support matrix to achieve a good seal. The paste on its own could be susceptible to extrusion through the joint due to pressure applied over time. Thread tapes or cord on their own can be subject to leakage through gaps in the matrix. A combination of the two has the ability to provide a good space filling seal and also resist extrusion providing a high resistance to pressure across a wide range of thread types and joint makeup.

Non-hardening jointing compounds appear to be more reliable when combined with a support substrate of some sort. None of the joints where support material was used in conjunction with compound developed any leakage. This includes a direct comparison with two of the same compounds that did develop leakage when used alone. The third compound also used with PTFE cord did not leak when used alone.

From a leak development perspective, there appears to be no difference within this scope whether the support is provided by PTFE cord, unsintered PTFE tape, or hemp.

Further investigation would be required to determine whether the issues found with some of the sealants is caused by hydrogen, or whether a similar trend would be observed with methane or Natural Gas.

9.3 Suitability of gas valves

Gas rated commodity ball valves showed a propensity to develop leaks through the valve stem seals when in an open position. The NBR seal material is generally considered as having excellent compatibility with hydrogen, and the valves were rated for the temperatures. It is unclear whether the unsuitability of the valves is related to hydrogen, or commodity gas valves in general. Anecdotal information provided by industry experts indicates that the valves are known to develop stem leaks with Natural Gas.

There is indication that the stem seals had hardened and suffered lowered elasticity, reducing their sealing properties. It is possible that the same result would be observed if similar experiments were carried out with Natural Gas or methane. Further investigation is recommended to determine the extent of this issue, and identify whether it is caused by hydrogen or presents an increase in hazard due to hydrogen.

9.4 Air ingress

Air ingress was observed to occur through small leaks at a greater rate than anticipated. Clearly this could lead to dangerous situations in dormant installations where pressure is allowed to decay. During this project an additional study was carried out to determine whether this was a problem specific to, or exacerbated by hydrogen. The results indicated that methane demonstrates the same effect, but at a slower rate. As a result of the findings from this project, a more detailed study into air ingress as a comparison with methane is recommended.

9.5 Cause of leakage development

This study demonstrated which sealant types and combinations did not develop leakage over a period of 41 weeks at room temperature, and at elevated temperatures simulating durations up to 50 years. Some sealants did develop leakage however, and the issue of compatibility and understanding of the reasons for failure remain. This project cannot determine whether the deterioration observed was caused by hydrogen. A further investigation would be required to make this assessment.

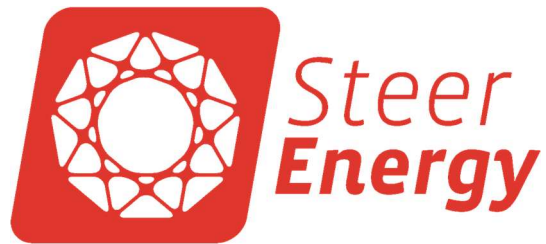
During development of the list of sealants to test in this project, some of the sealant manufacturers noted that some products were approved for use with hydrogen. A review of the techniques used was not carried out during this study, but the difference between comparative leak testing and testing for chemical compatibility was highlighted. It is unclear whether hydrogen chemical compatibility testing had been carried out on these sealants.

Due to the project constraints, control tests pieces with methane were not included. Further investigation is recommended to carry out equivalent testing in methane to help understand the differences in chemical compatibility, and whether hydrogen poses an additional risk in terms of leak development in sealants compared to Natural Gas.

10 Bibliography

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11 Appendix A – Test Cards



Sealant Suitability

5147/06/2021 (Var. 3)

Final Report v6.0

Appendix A – Test Cards:

**Anaerobic Jointing Compounds
(BS EN 751-1)**

Sealant Suitability

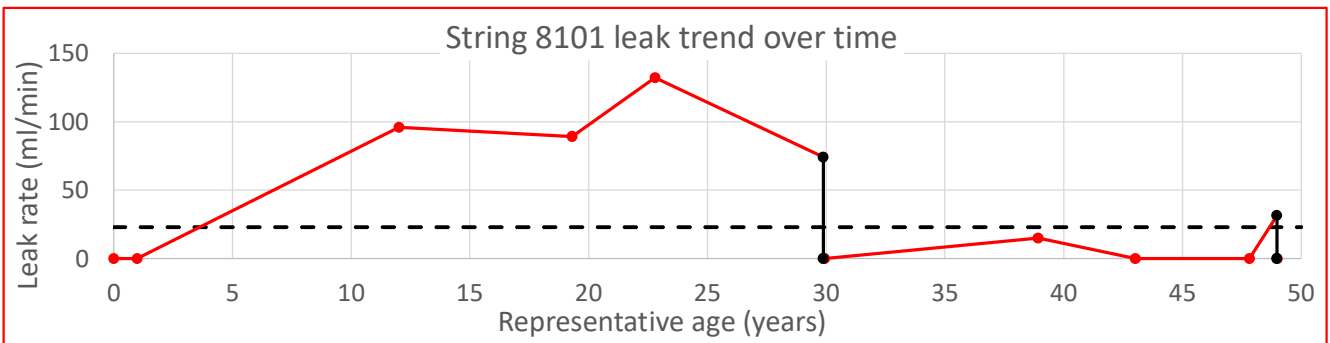
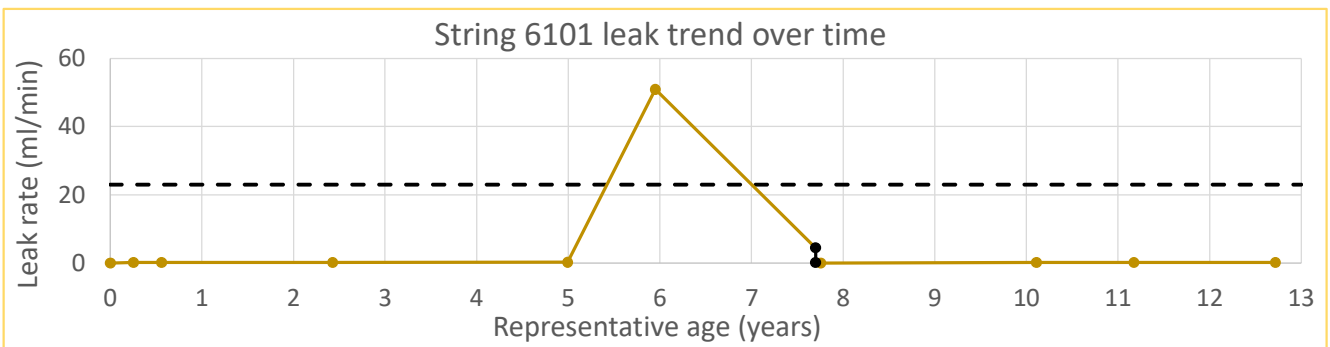
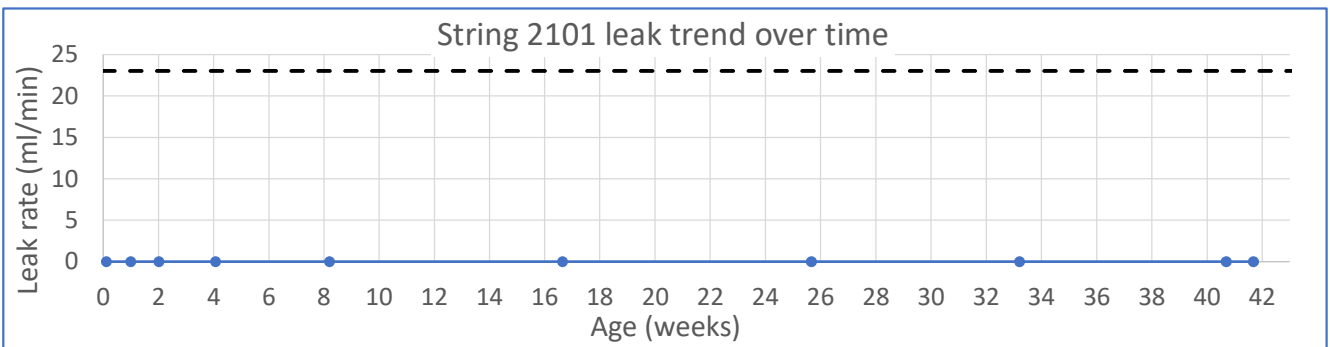


Sealant 101

Anaerobic jointing compound

String reference		
2101	6101	8101

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 101 passed with no leakage. The leakage seen in 6101 and 8101 was from Valve 1.

Sealant Suitability

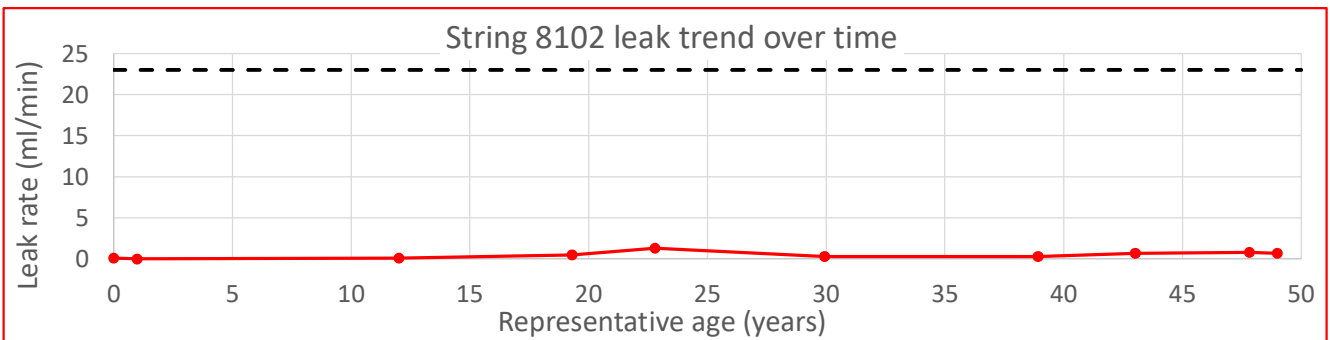
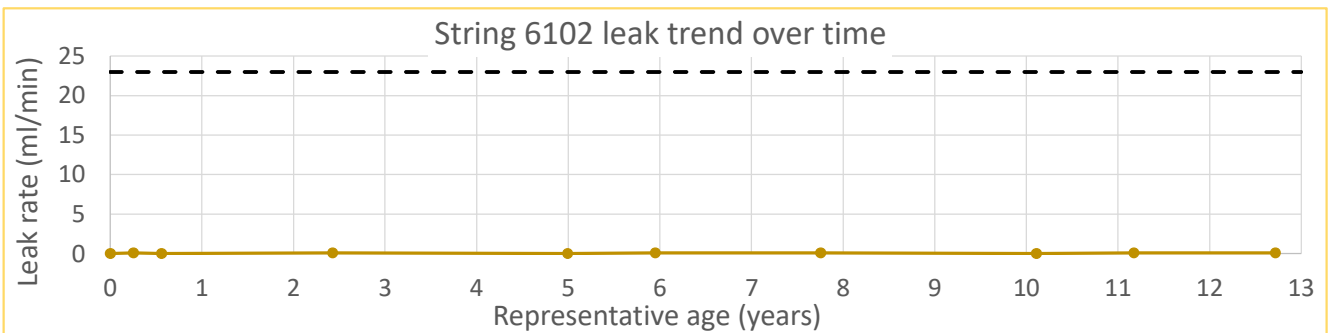
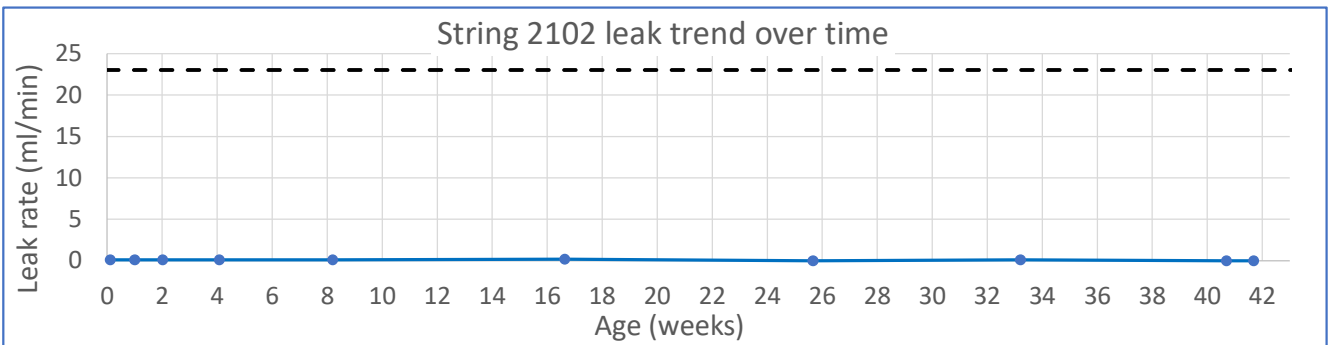


Sealant 102

Anaerobic jointing compound

String reference		
2102	6102	8102

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 102 passed with no leakage.

Sealant Suitability

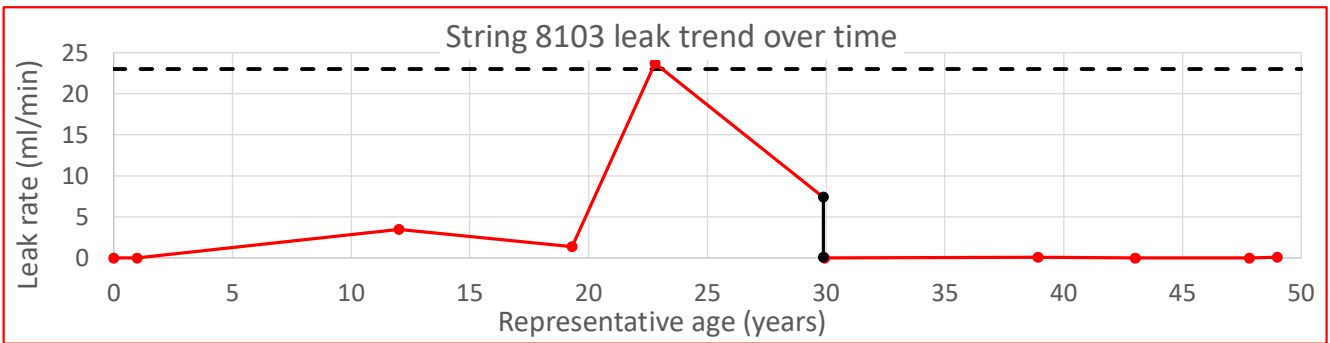
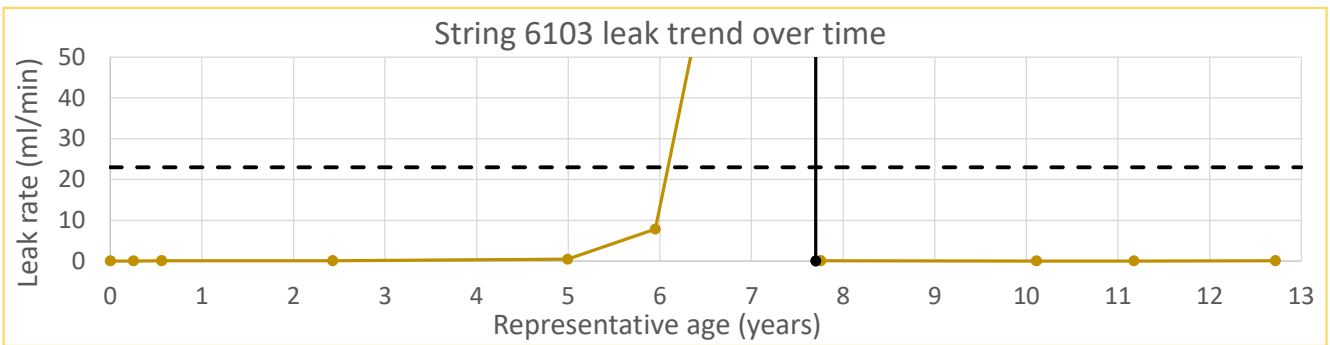
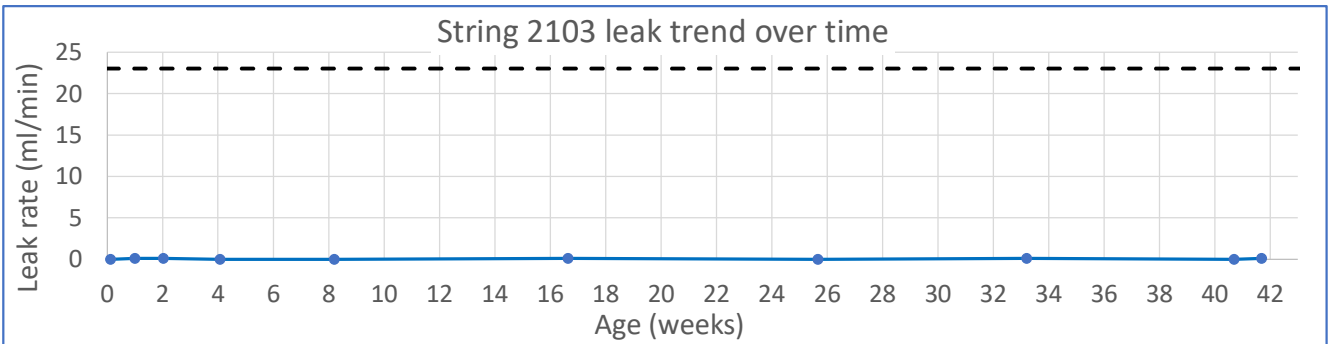


Sealant 103

Anaerobic jointing compound

String reference		
2103	6103	8103

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 103 passed with no leakage. The leakage seen in 6103 and 8103 was from Valve 1.

Sealant Suitability

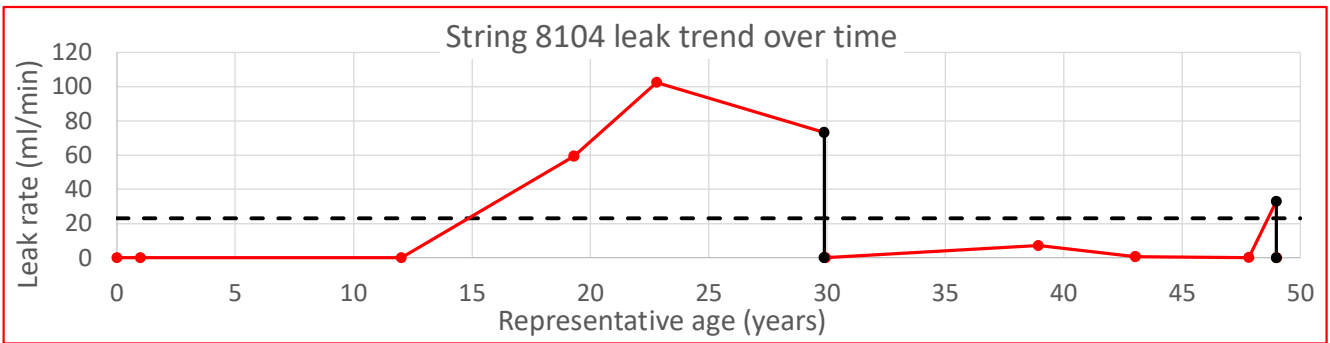
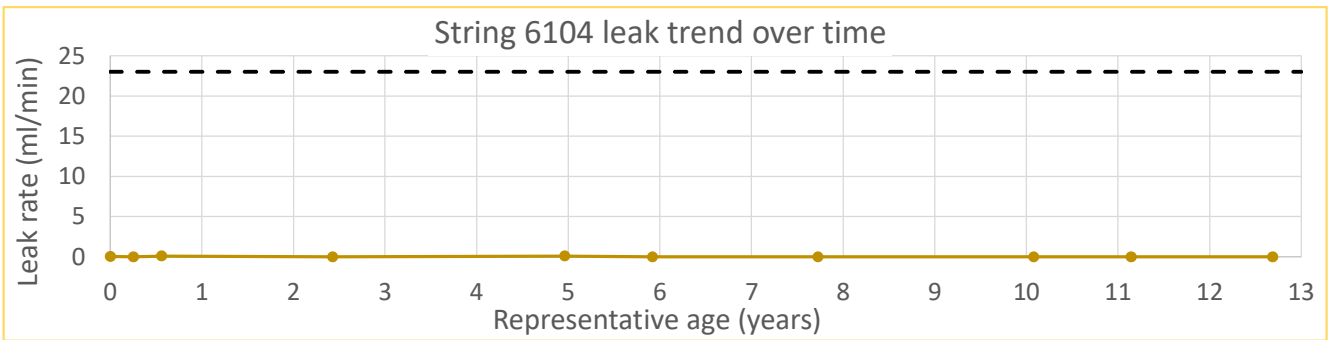
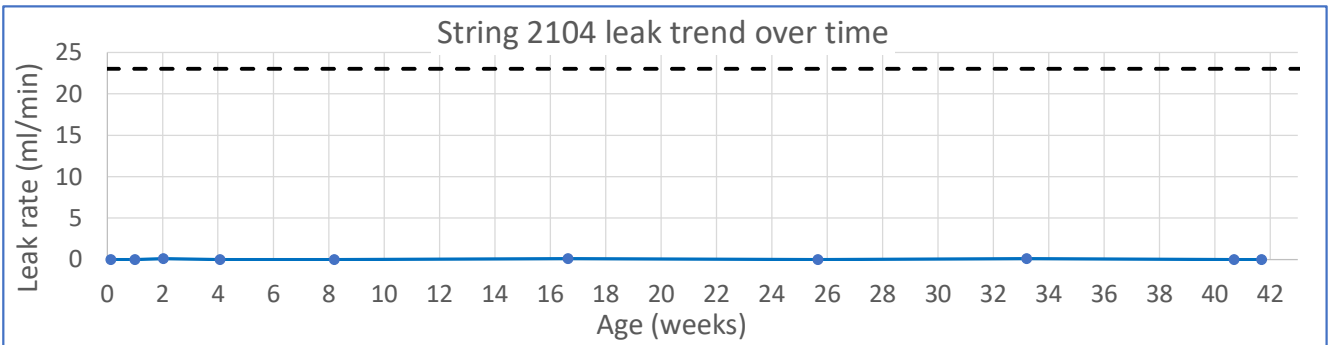


Sealant 104

Anaerobic jointing compound

String reference		
2104	6104	8104

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 104 passed with no leakage. The leakage seen in 8104 was from Valve 1.

Sealant Suitability

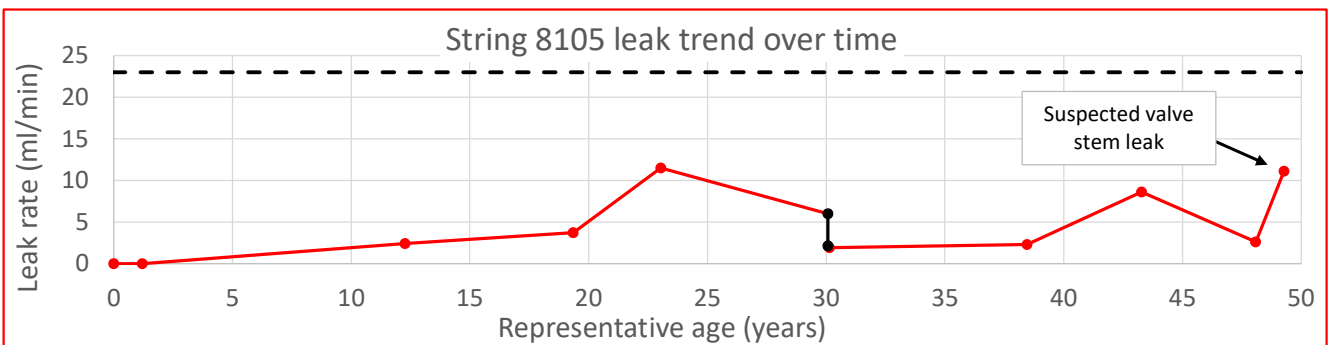
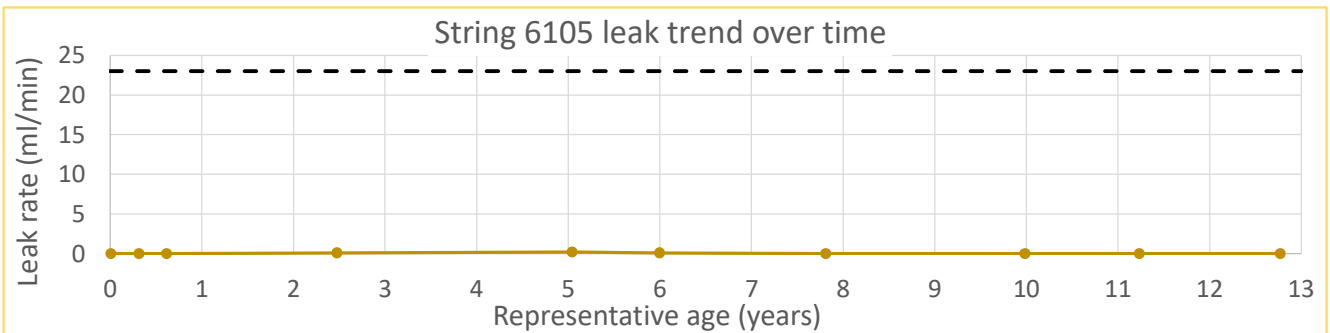
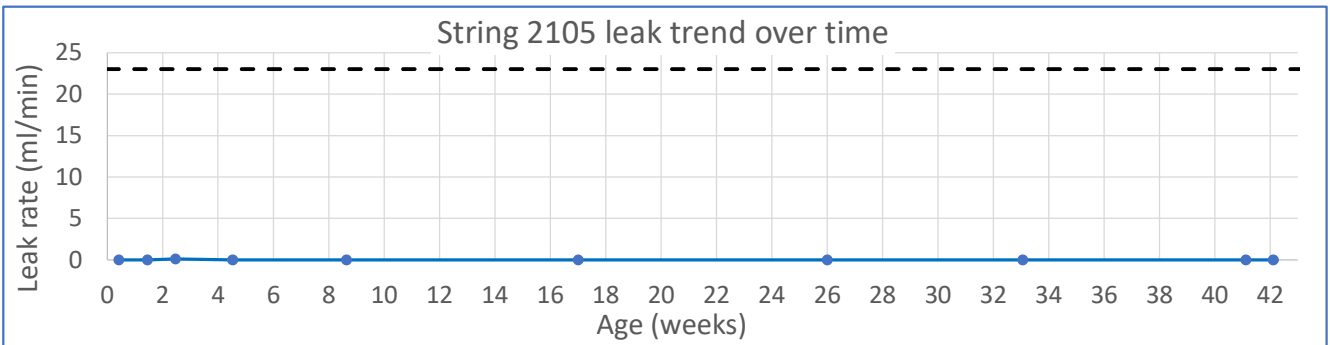


Sealant 105

Anaerobic jointing compound

String reference		
2105	6105	8105

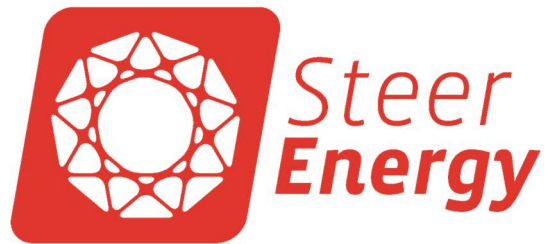
Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 105 passed with none to very little leakage. The majority of the leakage seen in 8105 was from Valve 1. The leakage in the final test was mostly from the stem of Valve 1, but it is expected that some was leakage through a joint.



Sealant Suitability

5147/06/2021 (Var. 3)

Final Report v6.0

Appendix A – Test Cards:

**Non-hardening Jointing Compounds with
Support
(BS EN 751-2)**

Sealant Suitability

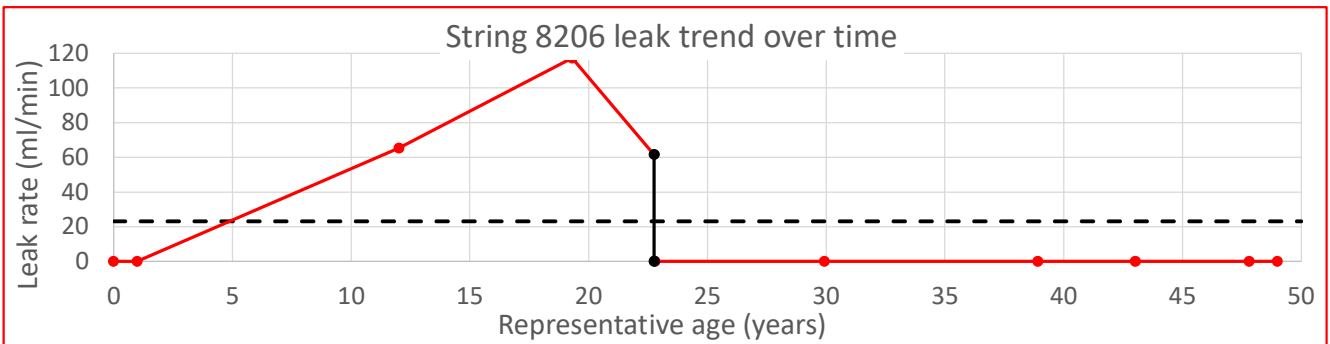
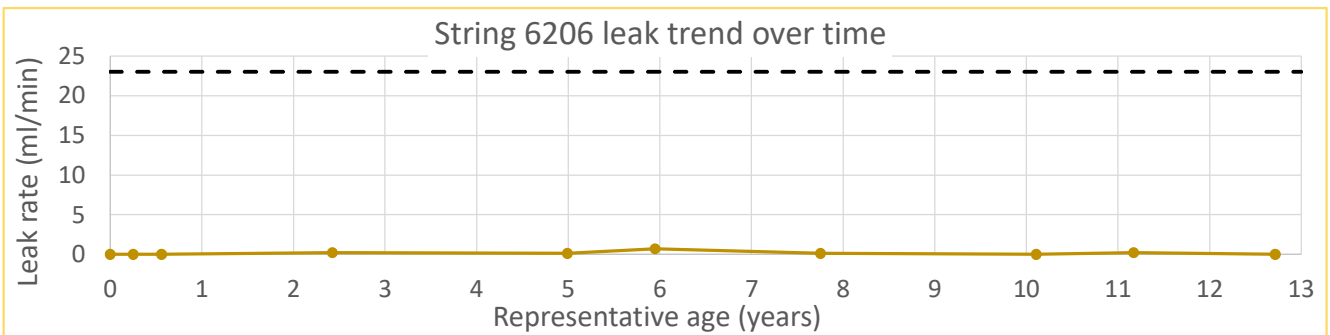
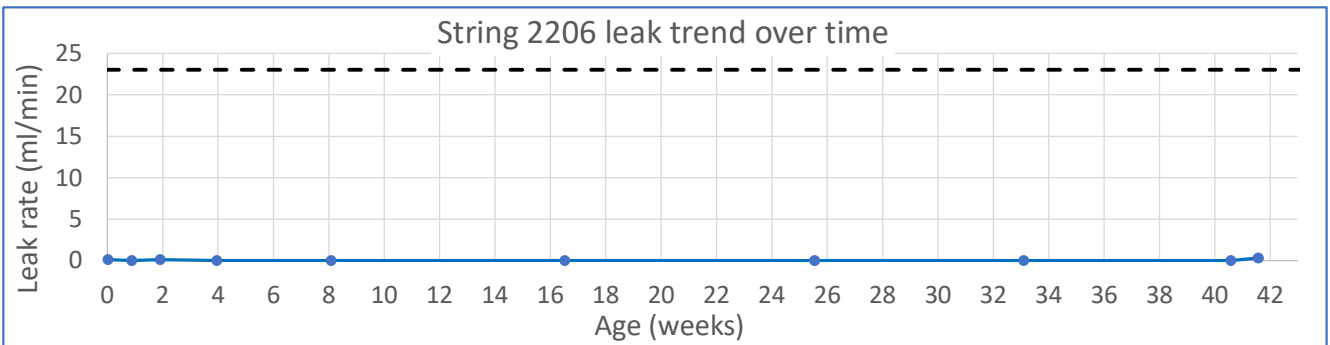


Sealant 206

Non-hardening jointing compound with support

String reference		
2206	6206	8206

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 206 passed and did not show any signs of leaks developing. Leakage observed in 8206 was from the stem of valve 1.

Sealant Suitability

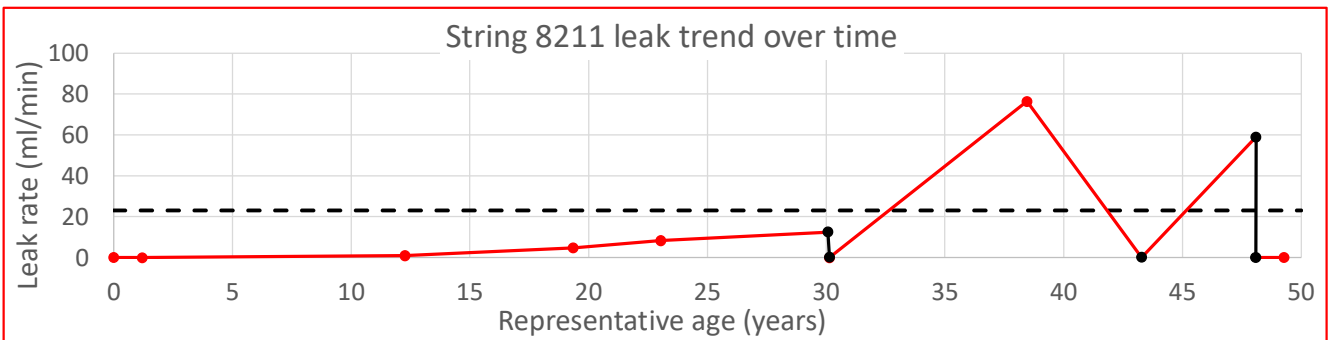
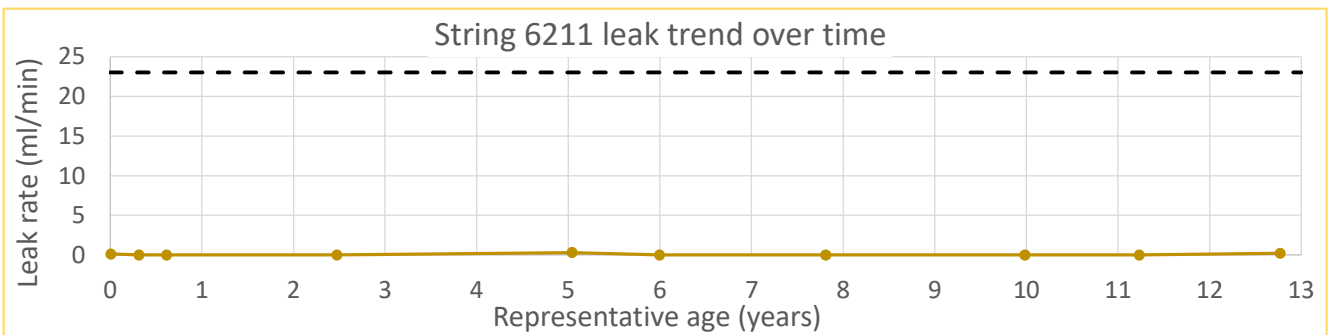
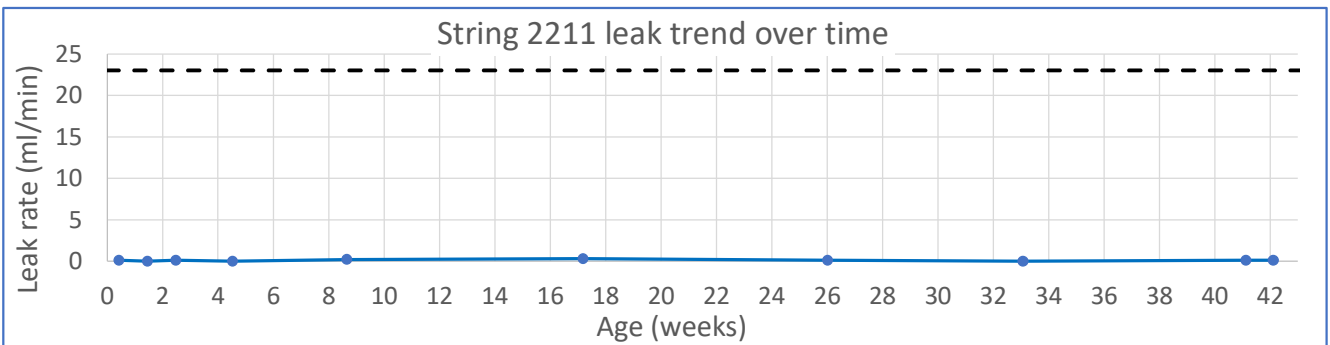


Sealant 211

Non-hardening jointing compound with support

String reference		
2211	6211	8211

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 211 passed with no leakage. Leaks observed from 8211 were from valve stems.

Sealant Suitability

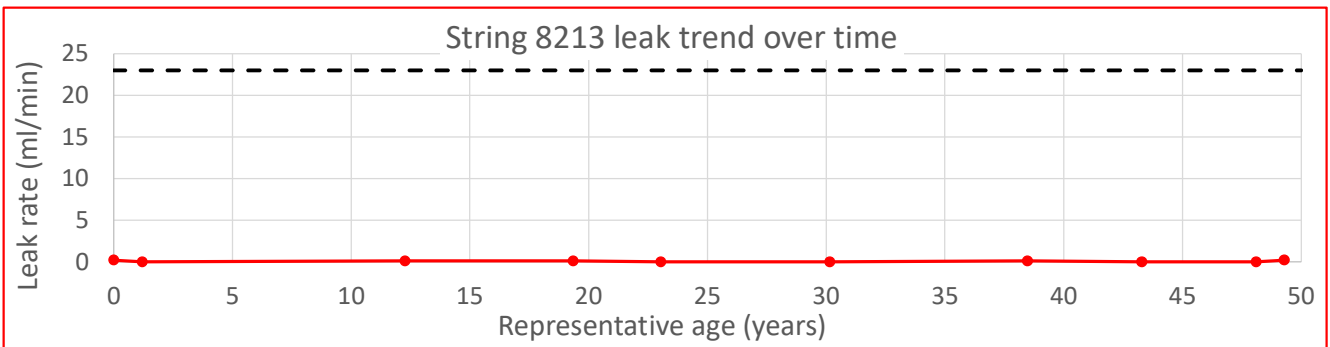
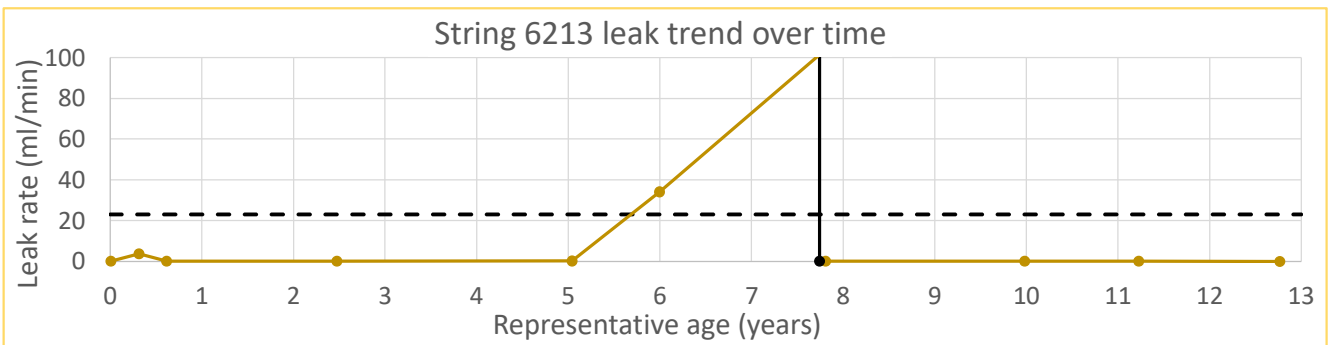
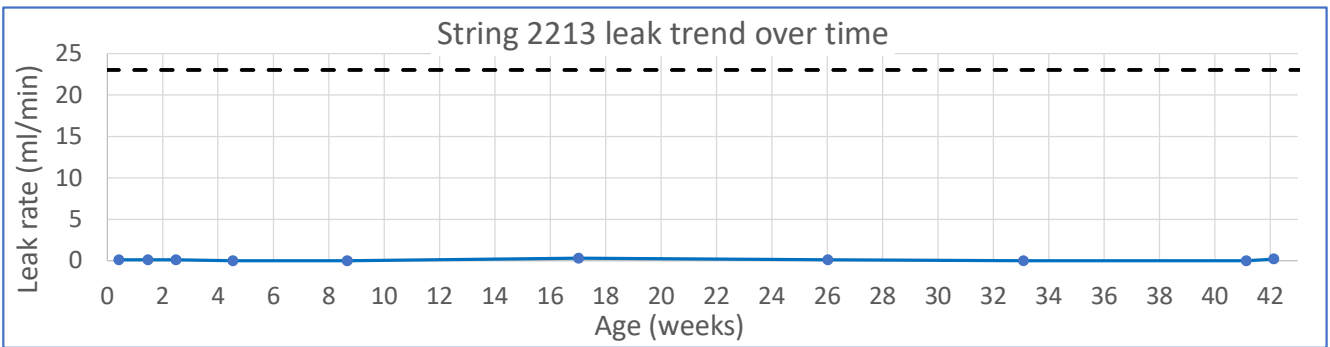


Sealant 213

Non-hardening jointing compound with support

String reference		
2213	6213	8213

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 213 passed with no leakage. Leakage in 6213 was from a valve stem.

Sealant Suitability

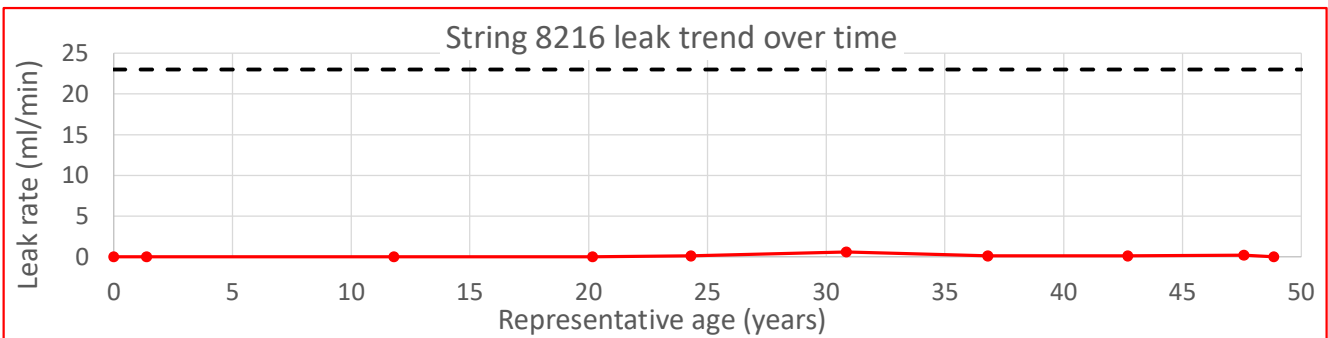
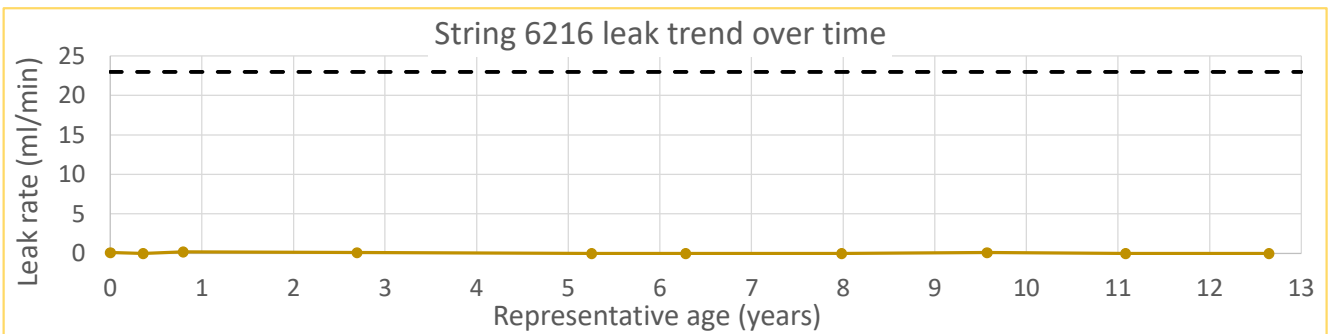
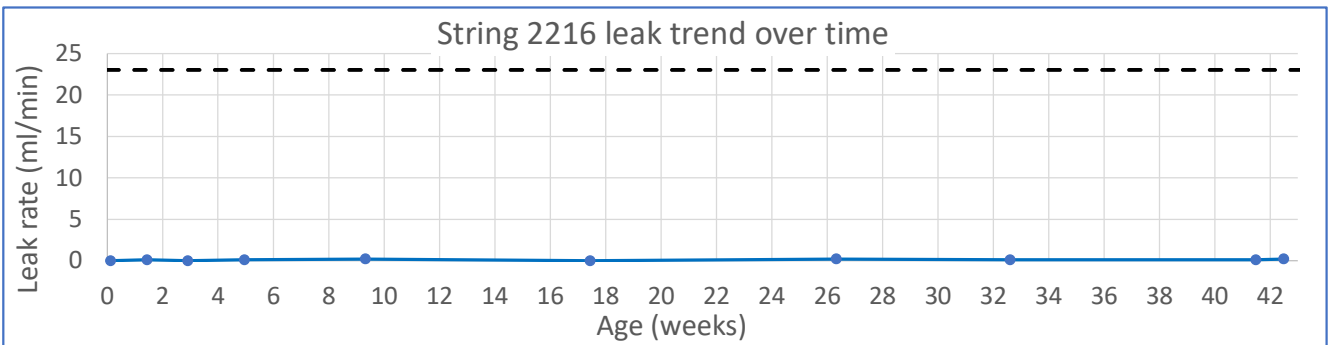


Sealant 216

Non-hardening jointing compound with support

String reference		
2216	6216	8216

Temperature of storage		
20°C	60°C	80°C

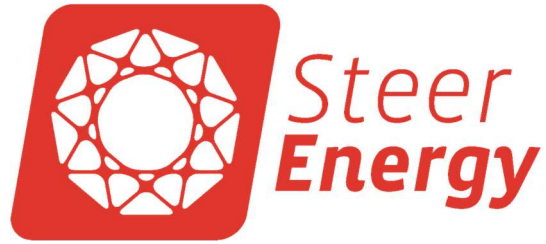


Legend

- Change due to valve replacement
- Valve replacement leak tests
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

Sealant 216 passed with no leakage.



Sealant Suitability

5147/06/2021 (Var. 3)

Final Report v6.0

Appendix A – Test Cards:

**Non-hardening Jointing Compounds
without Support
(BS EN 751-2)**

Sealant Suitability

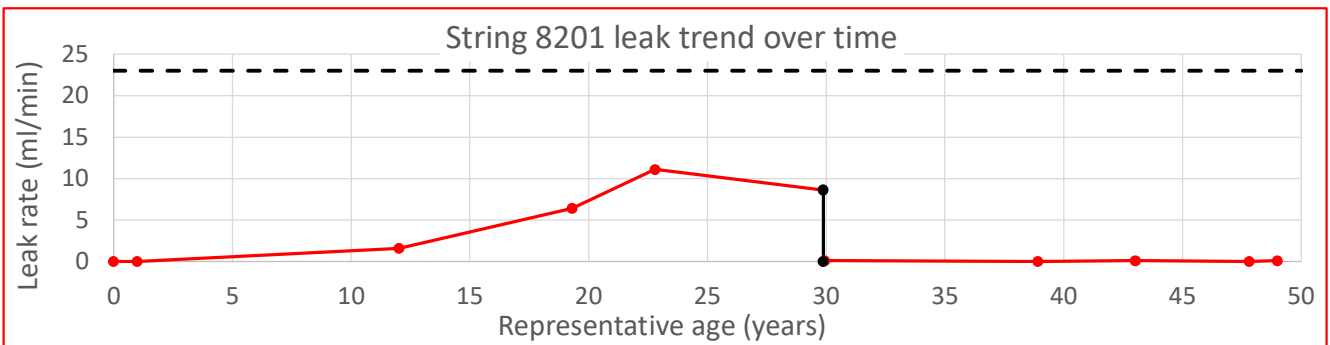
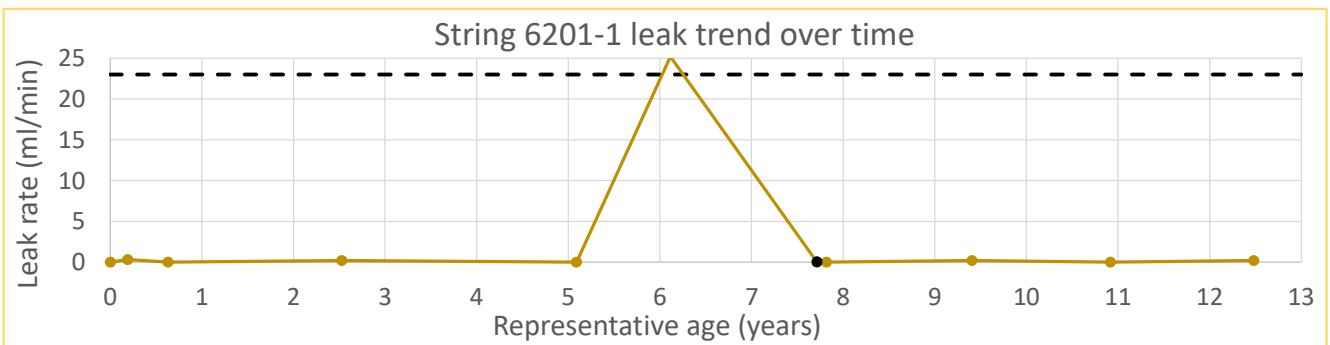
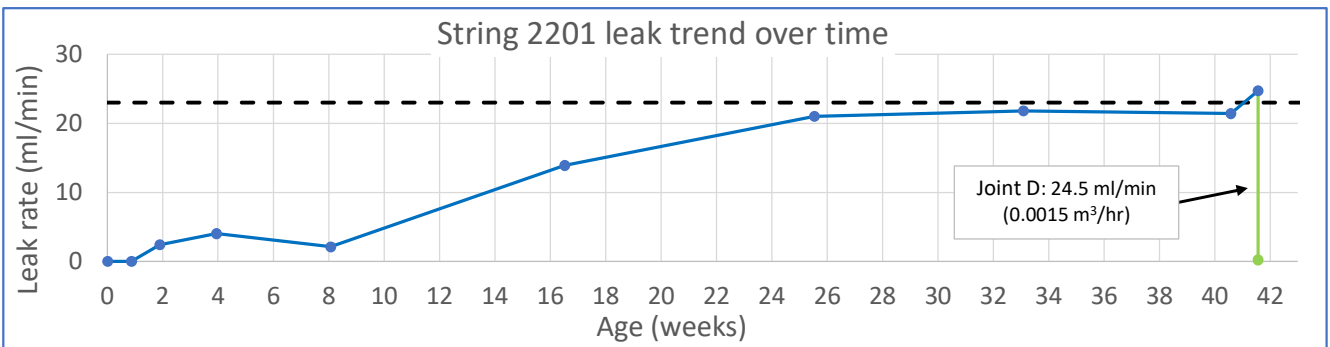


Sealant 201

Non-hardening jointing compound without support

String reference		
2201	6201-1	8201

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Retirement
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

Sealant 201 failed on joint D of 2201 with a leak rate of 24.5 ml/min (0.0015 m³/hr). The rest of string 2201 showed no leakage. 6201 failed after 1 week and was remade as 6201-1. The leakage in 6201-1 and 8201 was from the stem of valve 1.

Sealant Suitability

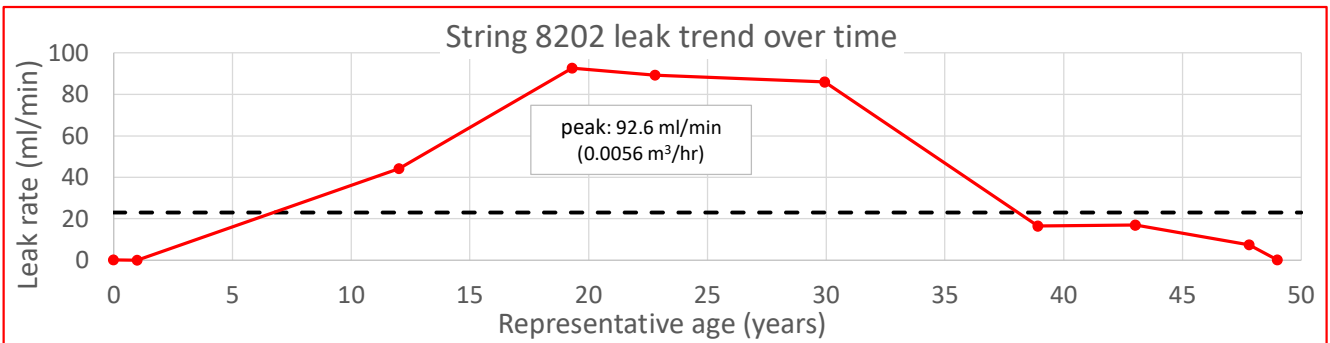
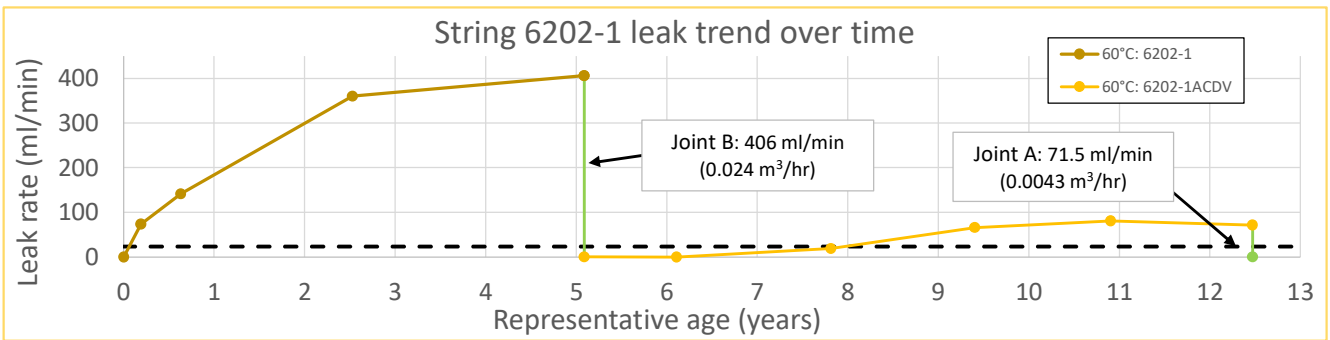
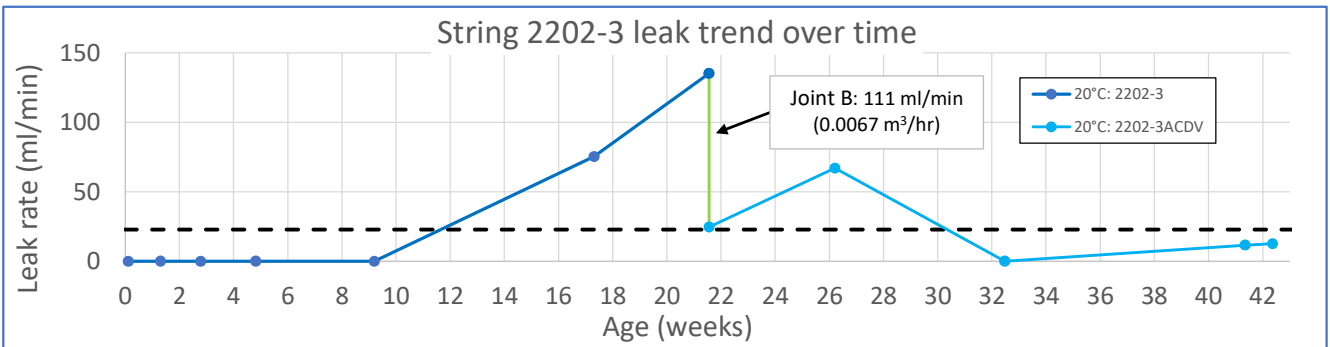


Sealant 202

Non-hardening jointing compound without support

String reference		
2202-3	6202-1	8202

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Retirement
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

2202 and 6202 both failed after week 1 and were remade. 2202 was remade three times to produce a leak-free string. One joint in 2202-3 and two in 6202-1 failed and were retired. 6202-1 developed the largest leak in the test programme, of 406 ml/min (0.024 m³/hr). 8202 developed a leak of 92.6 ml/min (0.0056 m³/hr) but this reduced to nothing without apparent interference. This is considered to be a failure.

Sealant Suitability

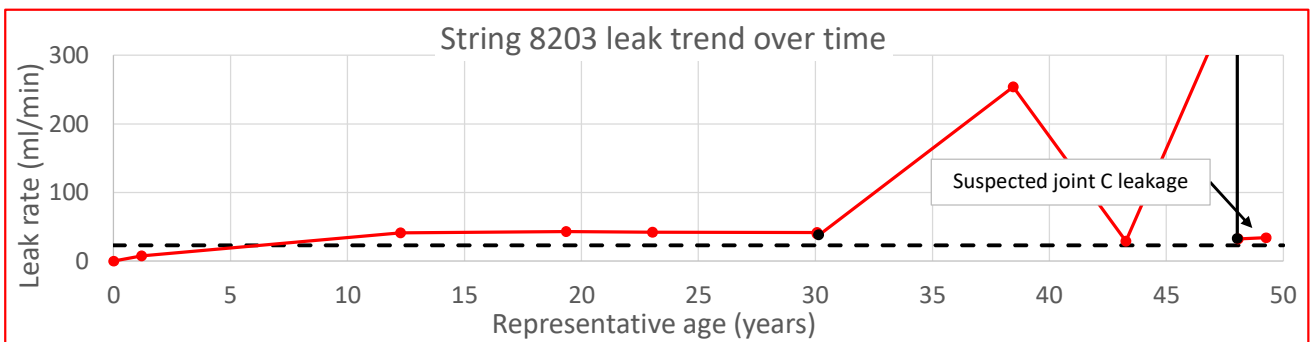
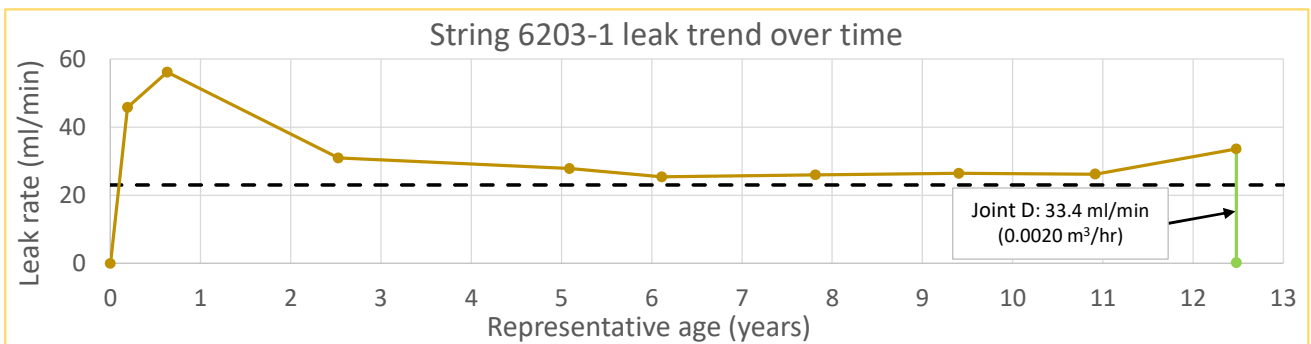
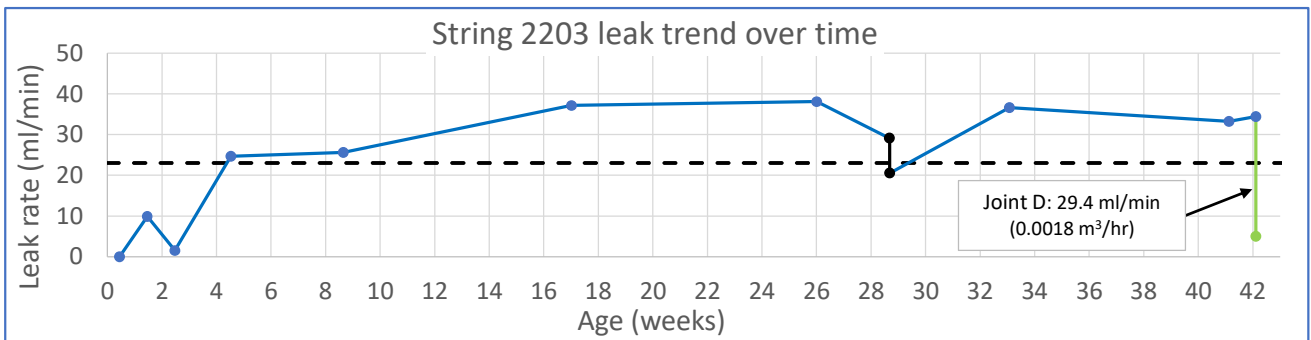


Sealant 203

Non-hardening jointing compound without support

String reference		
2203	6203-1	8203

Temperature of storage		
20°C	60°C	80°C



Legend			
Change due to valve replacement	Valve replacement leak tests	Retirement	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

6203 failed after week 1 and was remade.

All three strings developed failure leak rates over the course of the test programme. The leakage was mostly from joints D in 2203 and 6203-1. The peak observed in 8203 (up to 400 ml/min not shown) was due to valve stem leakage. Leakage during the final test was identified as being from joint C. Sealant in 8203 cured hard and a final joint retirement to measure individual joint leak rates was not done.

Sealant Suitability

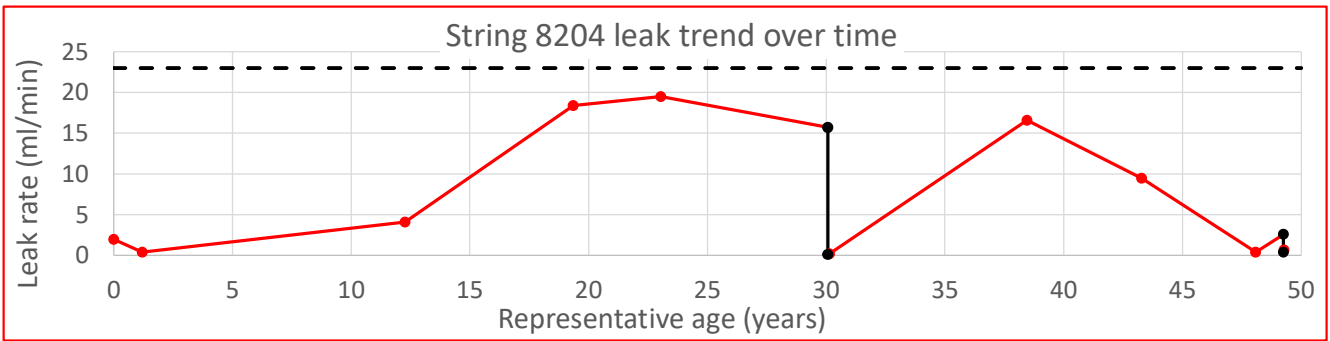
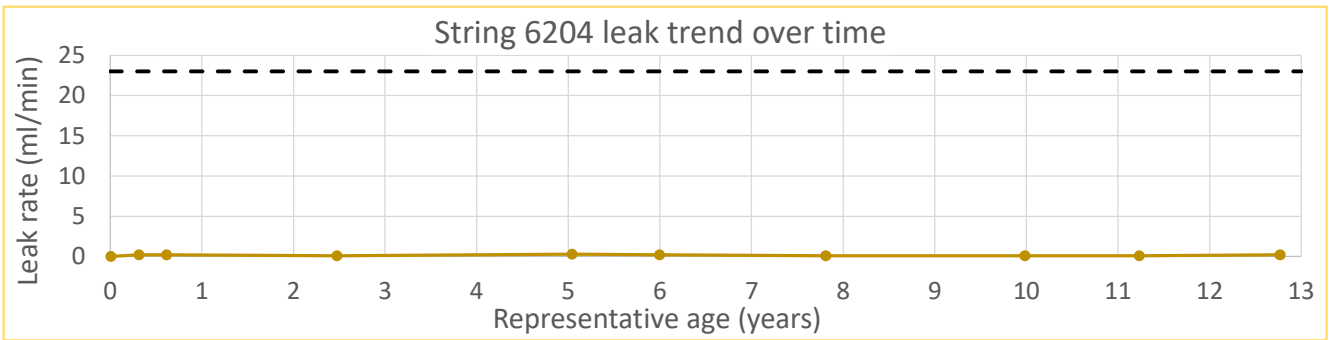
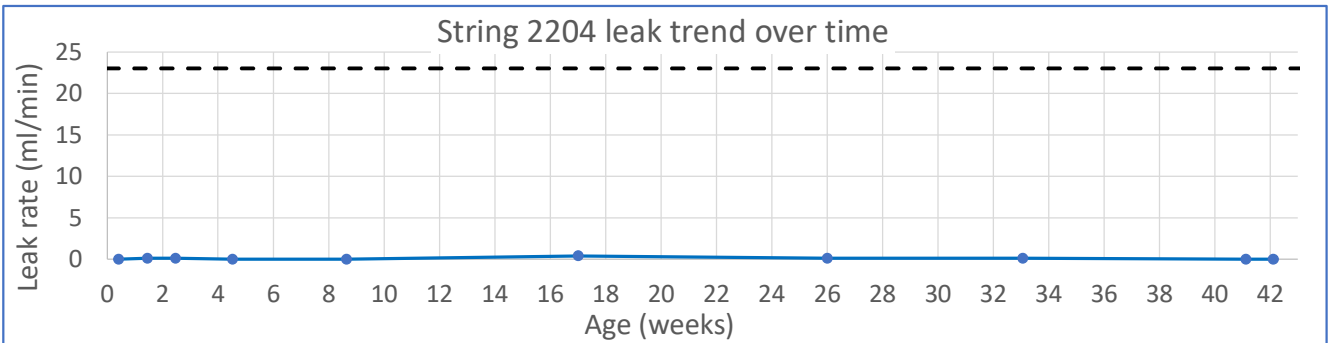


Sealant 204

Non-hardening jointing compound without support

String reference		
2204	6204	8204

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 204 passed with no leakage. The leaks in 8204 were valve stem leaks.

Sealant Suitability

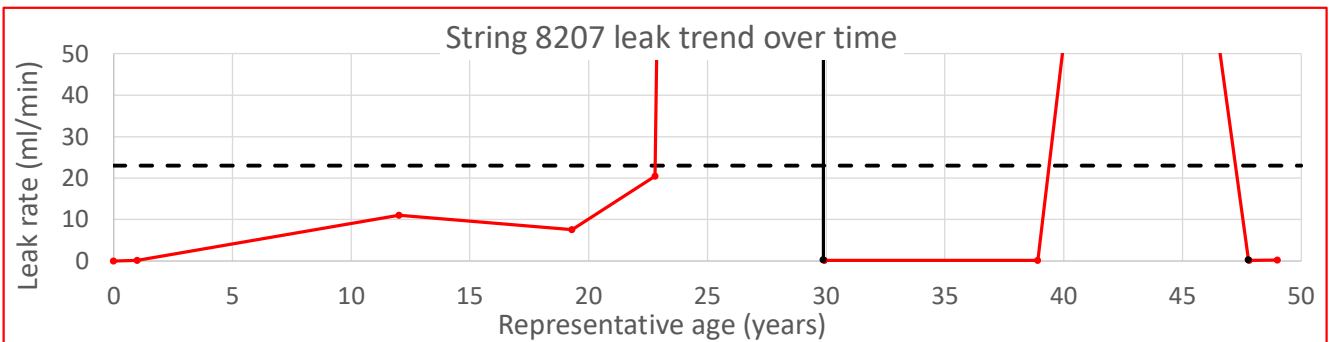
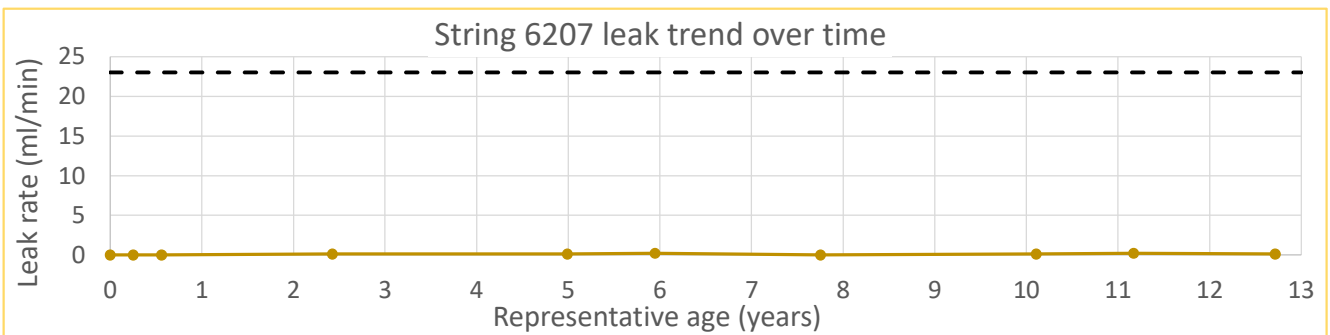
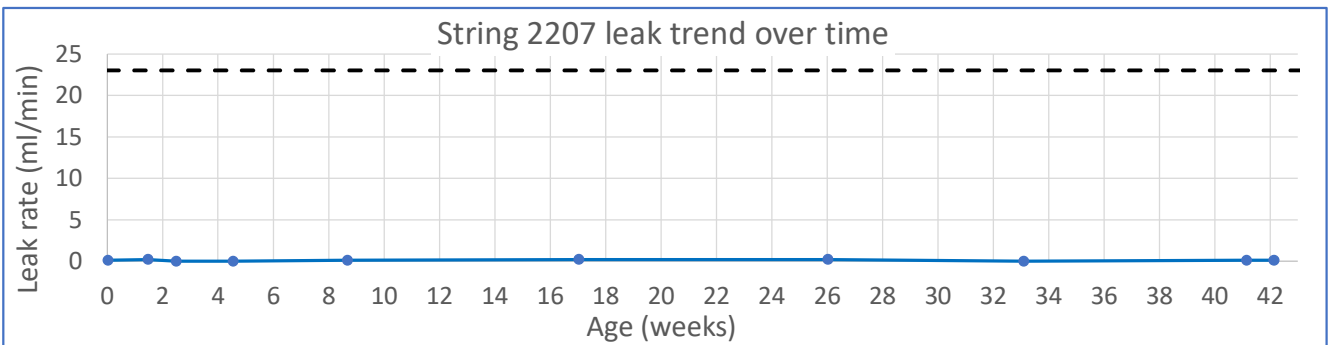


Sealant 207

Non-hardening jointing compound without support

String reference		
2207	6207	8207

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 207 passed with no leakage. Leakage recorded from 8207 peaked at 3,000 ml/min (0.18 m³/hr) and was from the stem of valve 1. Leakage of 198.6 ml/min (0.012 m³/hr) from 8207 at 43 years was temporary and believed to be from a valve stem.

Sealant Suitability

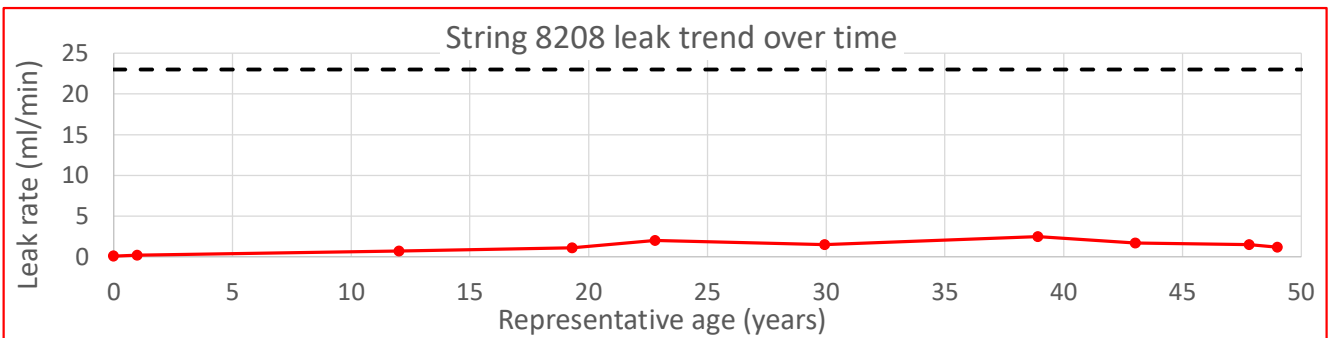
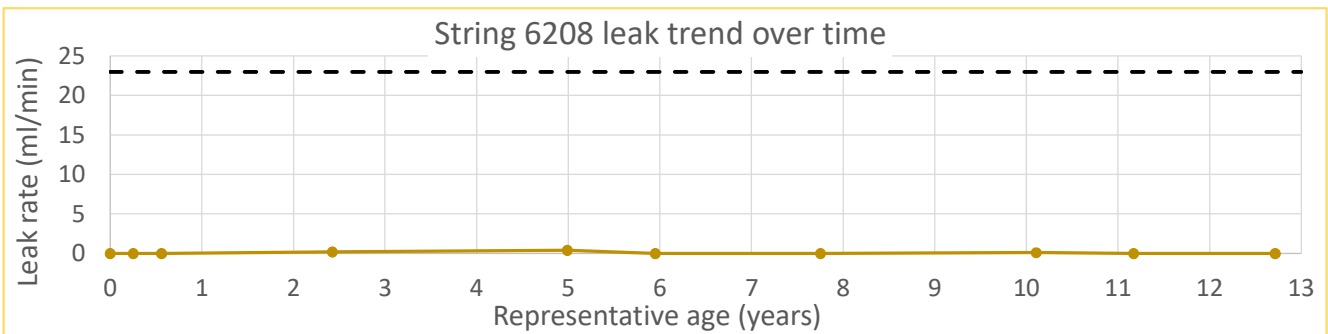
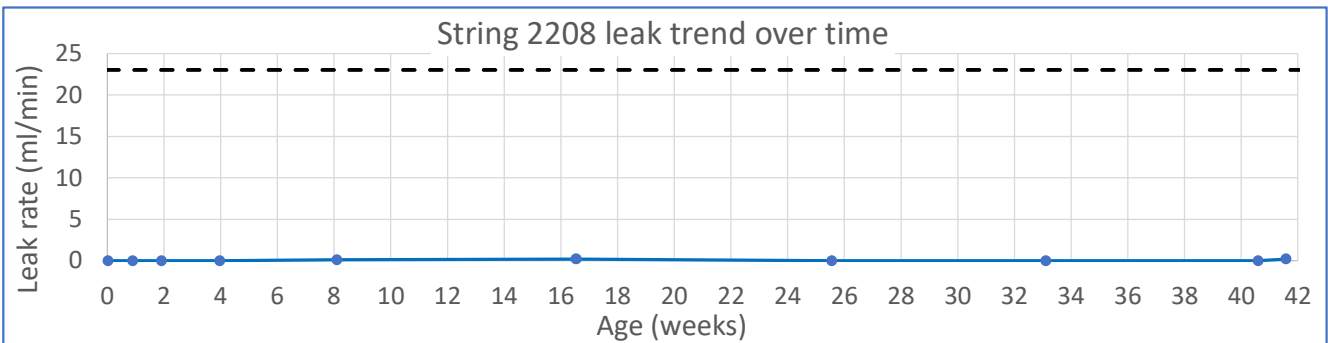


Sealant 208

Non-hardening jointing compound without support

String reference		
2208	6208	8208

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

Sealant 208 passed with no leakage from 2208 and 6208, while leakage from 8208 was minimal. It is unknown whether 8208 leakage was from a valve stem or test joints.

Sealant Suitability

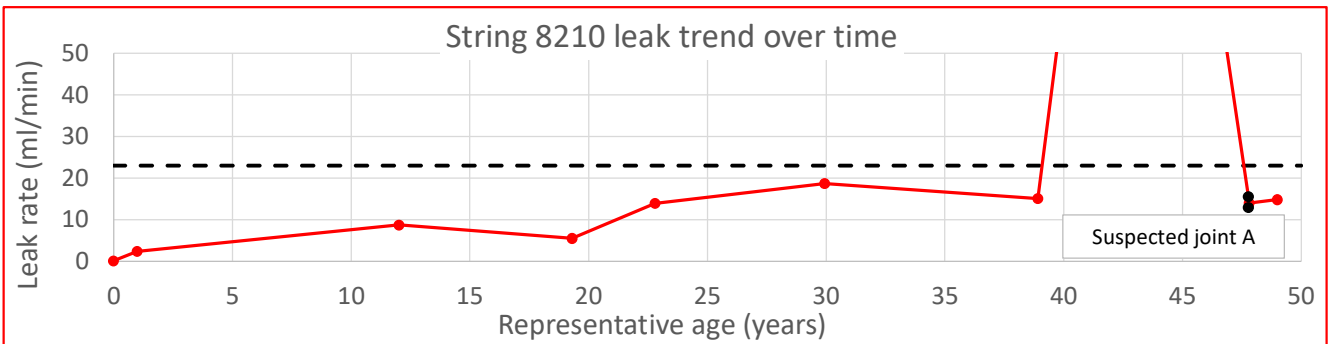
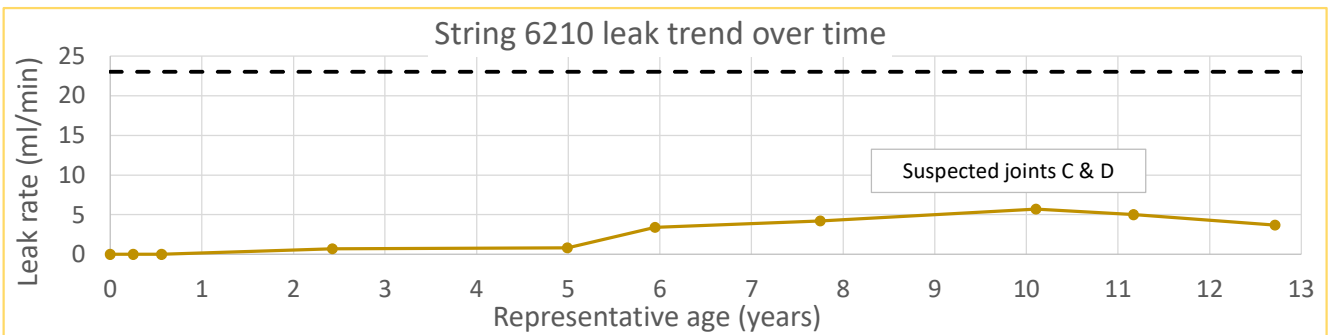
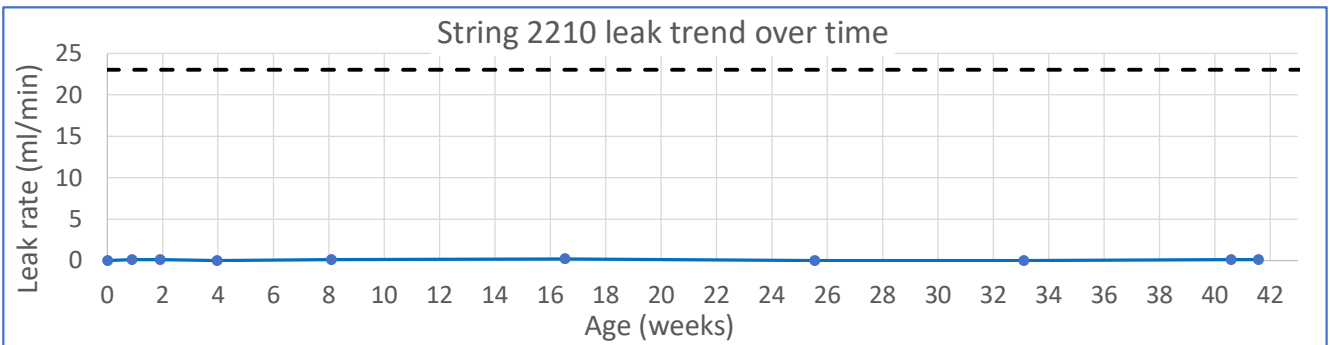


Sealant 210

Non-hardening jointing compound without support

String reference		
2210	6210	8210

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

All three sealant 210 strings passed, however 6210 and 8210 developed leaks. A spike of 198.7 ml/min (0.012 m³/hr) from 8210 was mostly from a valve stem, the remainder suspected to be from joint A.

Sealant Suitability

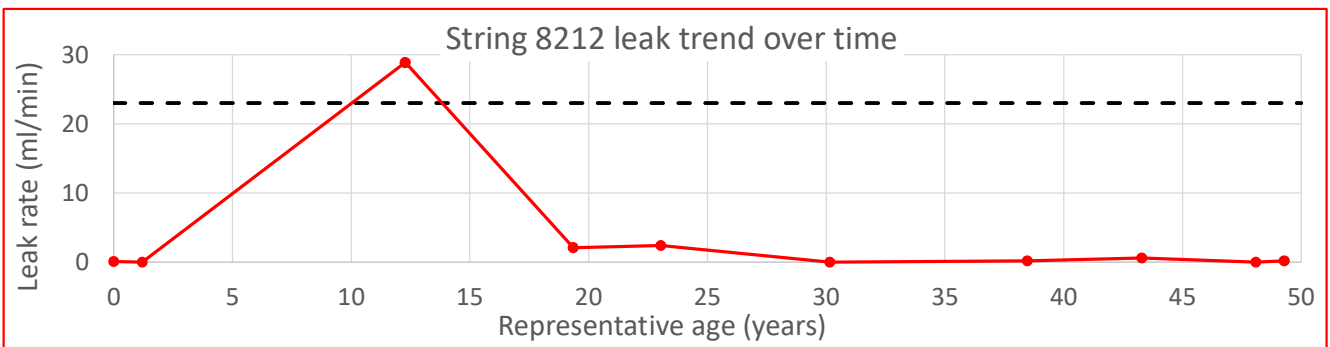
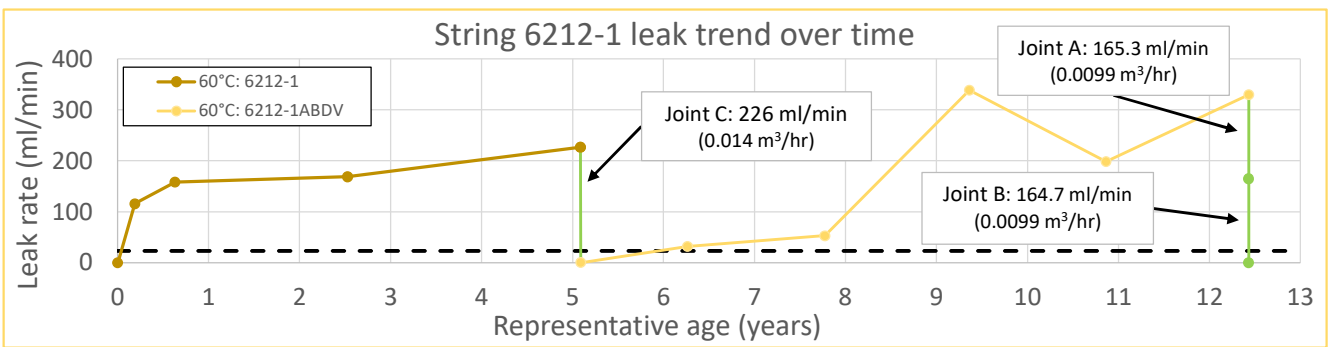
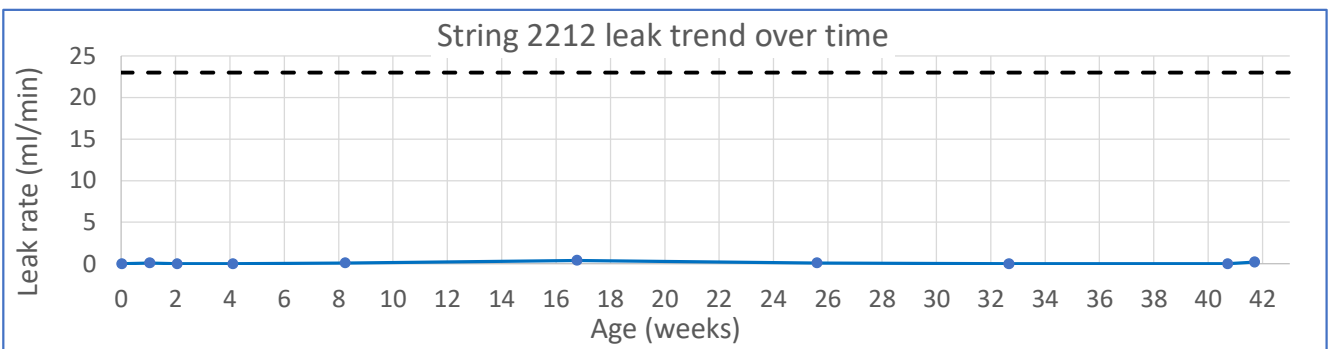


Sealant 212

Non-hardening jointing compound without support

String reference		
2212	6212-1	8212

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Retirement
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

Two strings of sealant 212 passed with no significant leakage. 8212 leaked temporarily for one test, the cause or location is not known, but the profile is typical of a vale stem leak.

6212 leaked after week 1 and was remade. 6212-1 also developed a leak and joint C was retired with a leak rate of 226 ml/min (0.014 m³/hr). The remaining string then developed final leak rates of 165 ml/min from A and 165 ml/min from B (both 0.0099 m³/hr).

Sealant Suitability

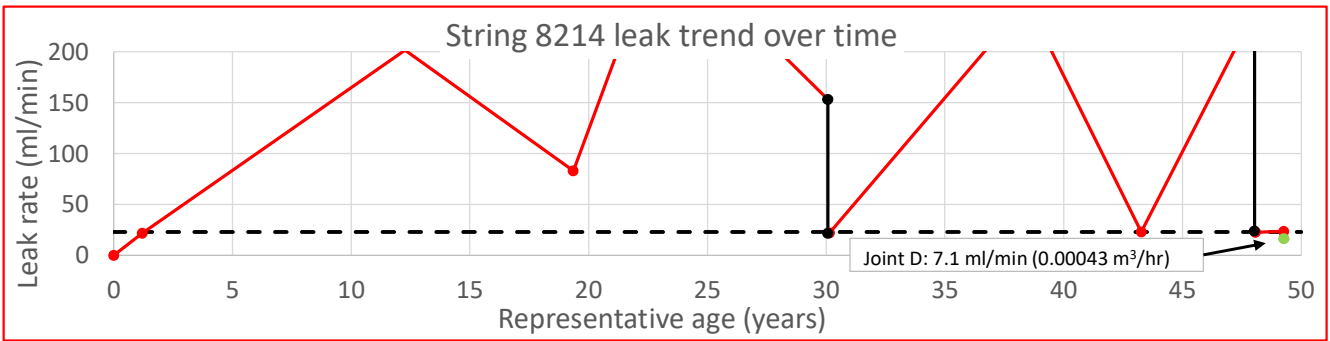
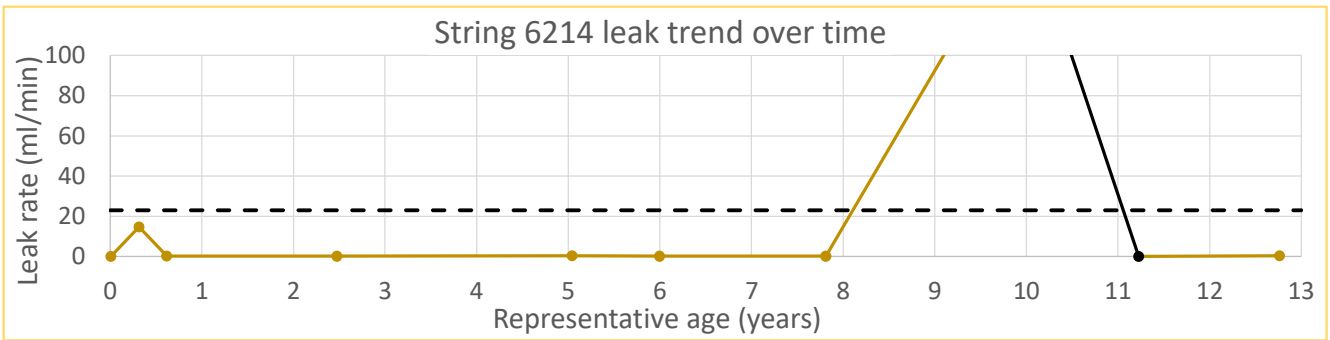
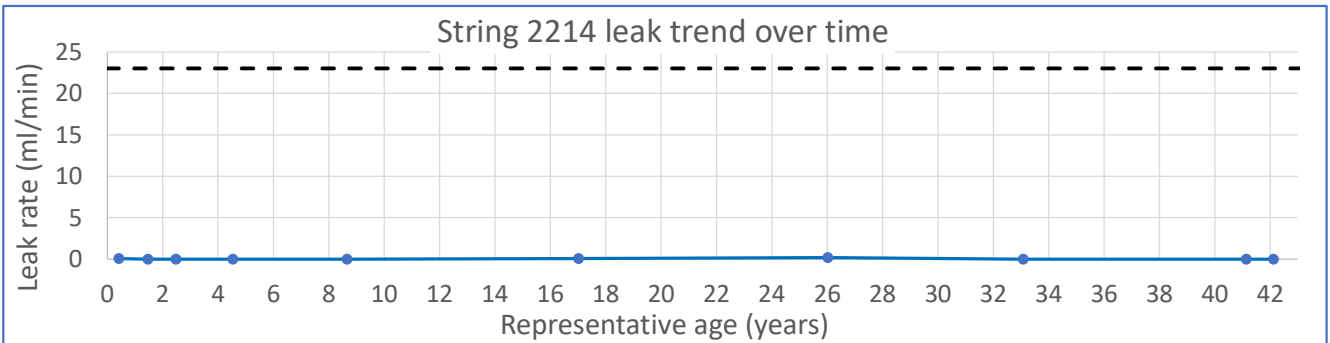


Sealant 214

Non-hardening jointing compound without support

String reference		
2214	6214	8214

Temperature of storage		
20°C	60°C	80°C



Legend			
Change due to valve replacement	Valve replacement leak tests	Retirement	Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

Sealant 214 had no leakage from 2214 or 6214. 8214 had leakage from valve stems, spiking up to 240 ml/min (0.014 m³/hr). 8214 had a final marginal failure leak rate of 23.5 ml/min (0.0014 m³/hr), of which 7.1 ml/min (0.00043 m³/hr) was attributed to joint D. None of the individual joints in 8214 had a large enough leak rate to fail.

Sealant Suitability

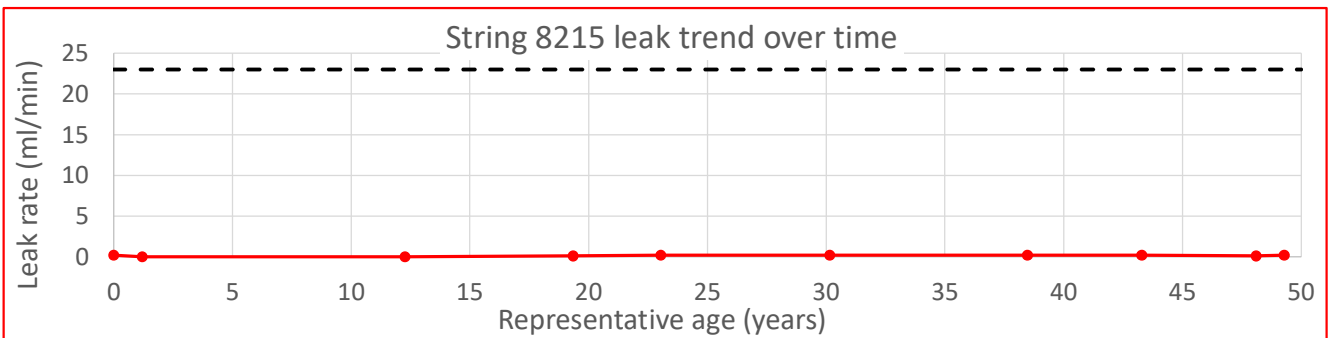
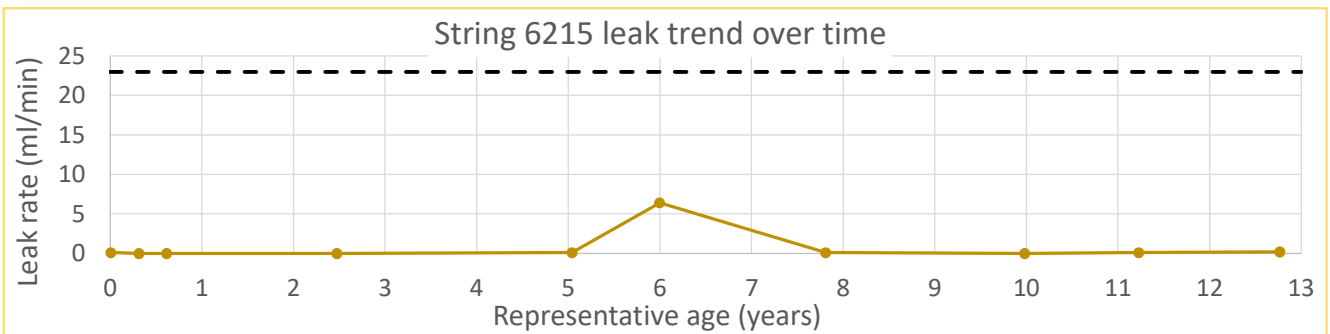
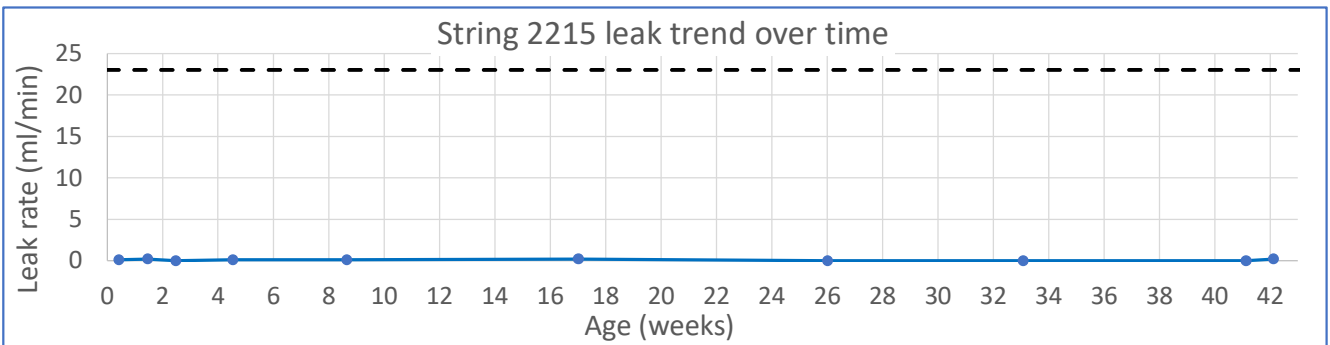


Sealant 215

Non-hardening jointing compound without support

String reference		
2215	6215	8215

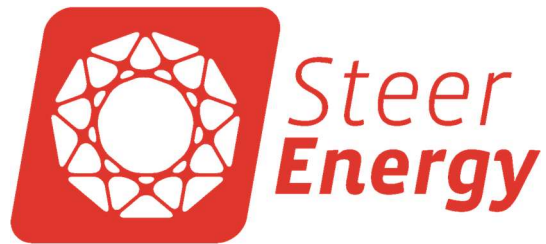
Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 215 passed with no significant leakage.



Sealant Suitability

5147/06/2021 (Var. 3)

Final Report v6.0

Appendix A – Test Cards:

**Unsintered PTFE Tape and Cord (String)
(BS EN 751-3)**

Sealant Suitability

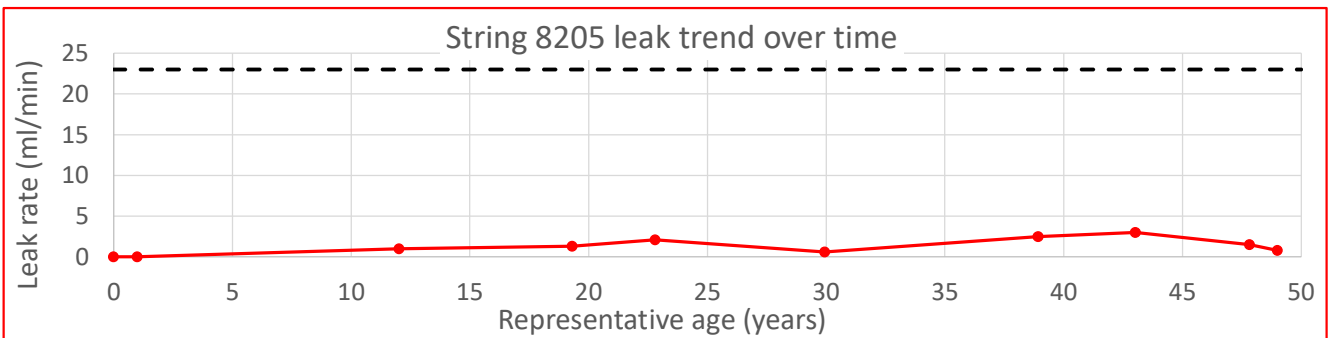
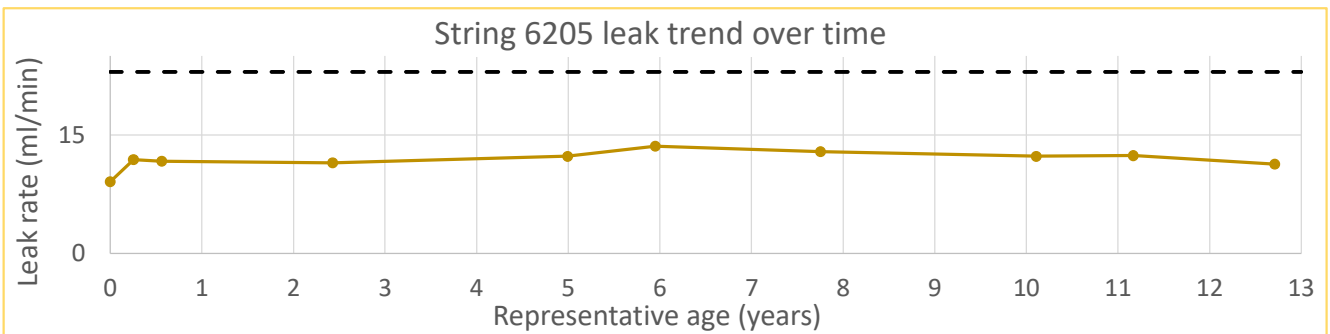
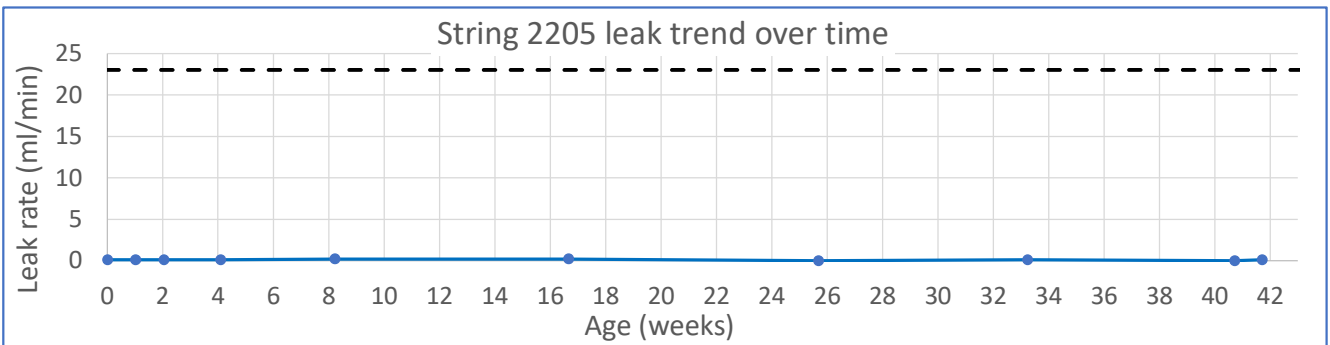


Sealant 205

Unsintered PTFE cord

String reference		
2205	6205	8205

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

No significant increase in leakage was observed with sealant 205. 6205 had some leakage from initial assembly. 8205 showed signs of leakage during the test programme but this did not develop into a significant leak.

Sealant Suitability

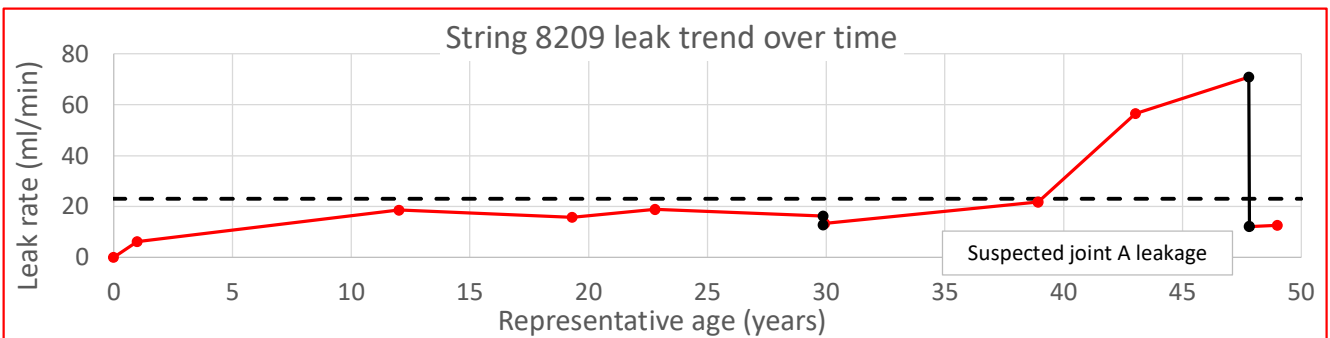
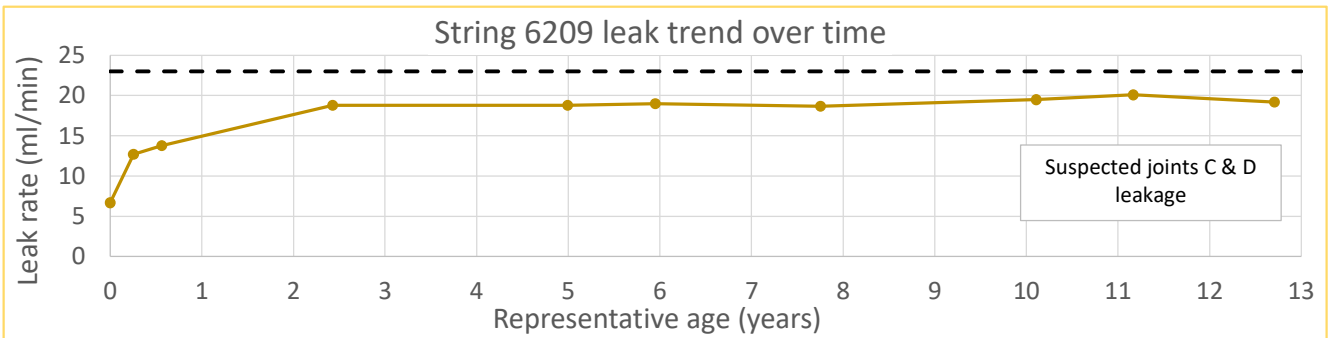
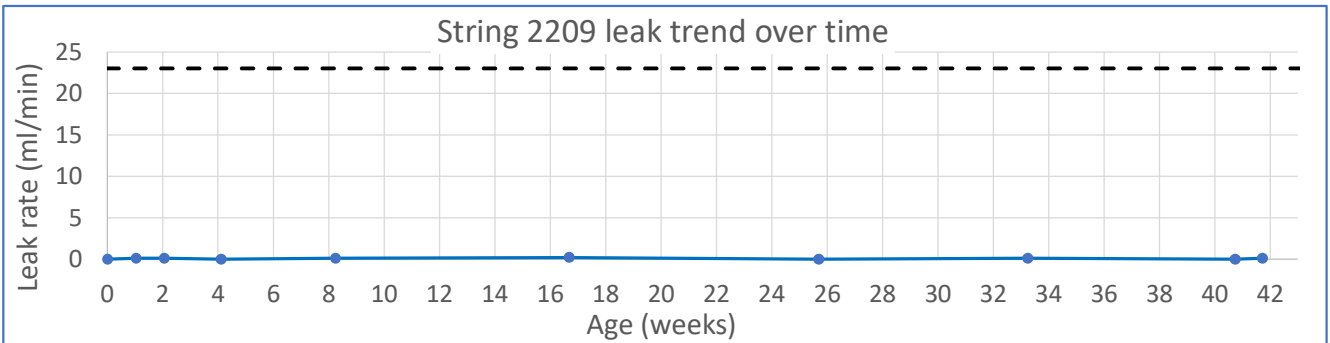


Sealant 209

Unsintered PTFE cord

String reference		
2209	6209	8209

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Sealant 209 developed some leakage in 6209 and 8209. Neither was beyond the failure leak rate, and 6209 had a baseline leak rate of 6.7 ml/min, indicating an increase of 13.4 ml/min (0.00080 m³/hr). This appeared to be mostly from joint C, with some from D. The leakage from 8209 after 40 years was from a valve stem, while the final leakage appeared to be from joint A. 2209 showed no sign of leakage.

Sealant Suitability

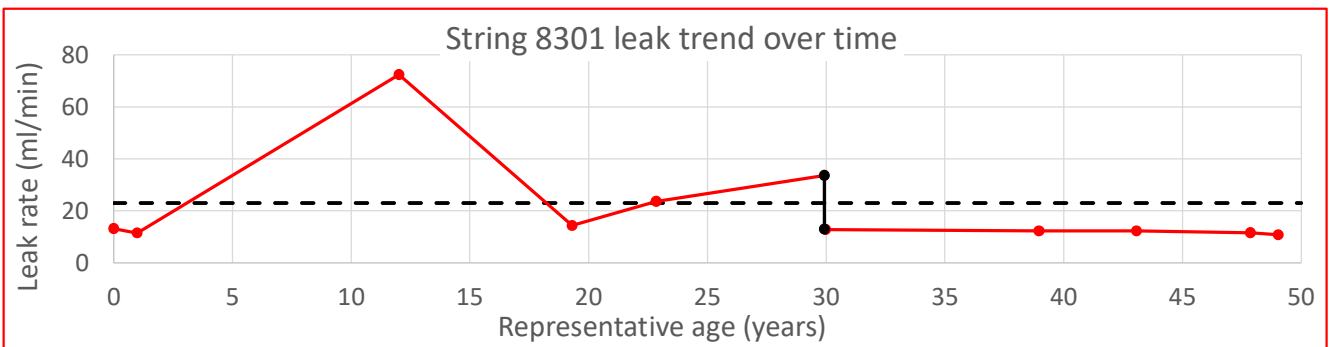
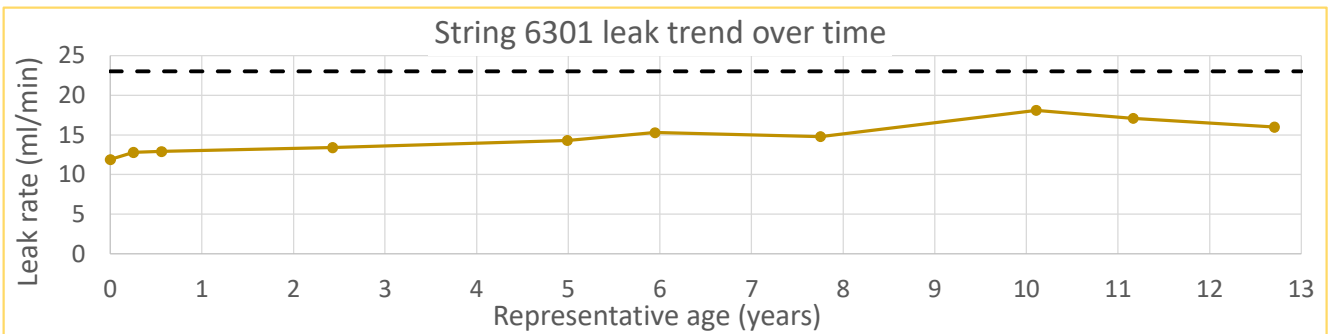
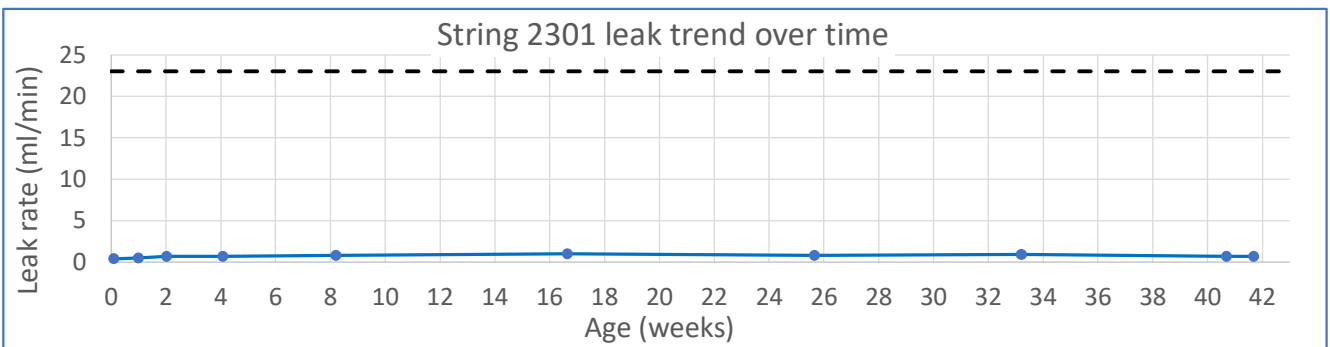


Sealant 301

Unsintered PTFE Tape

String reference		
2301	6301	8301

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

All three thick PTFE tape (yellow) strings showed some leakage from the initial assembly, though below the failure rate. Leakage from the 303 strings did not significantly increase over time. The spike of 72.4 ml/min (0.0043 m³/hr) from 8301 is likely but not confirmed to be from a valve stem.

Sealant Suitability

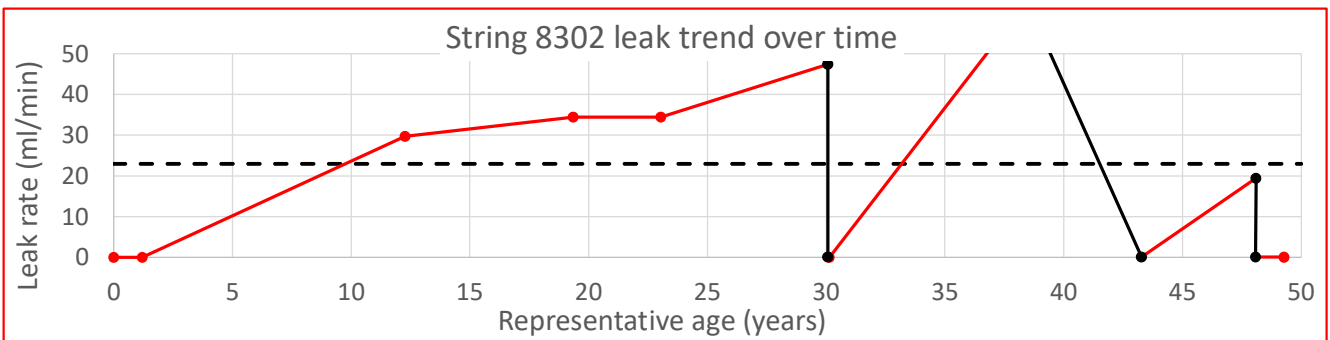
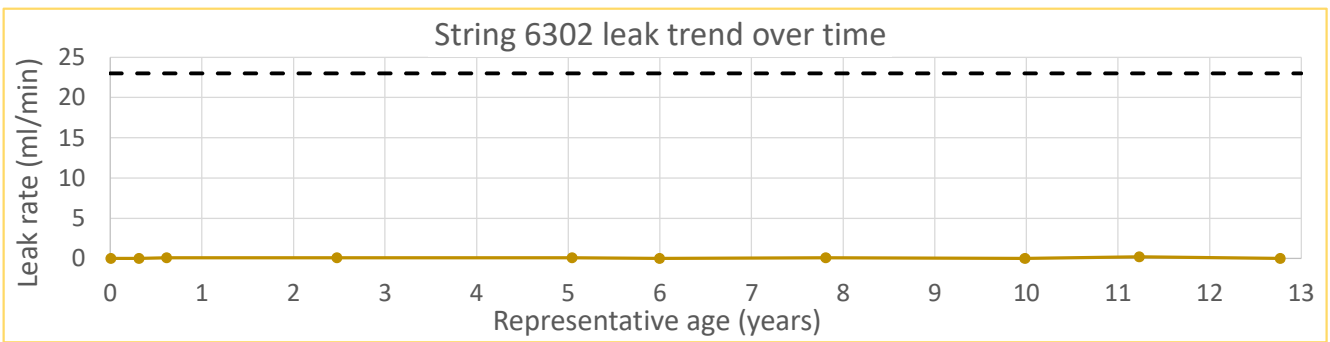
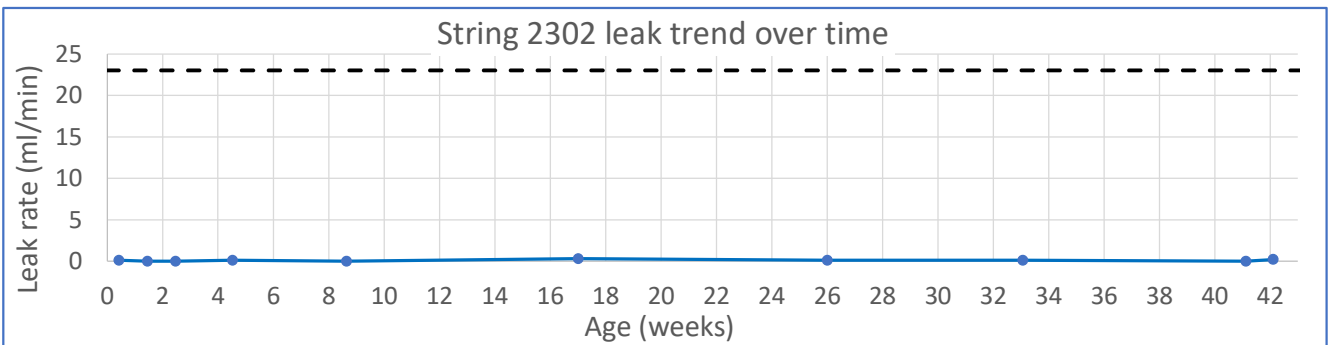


Sealant 302

Unsintered PTFE Tape & Non-hardening jointing compound

String reference		
2302	6302	8302

Temperature of storage		
20°C	60°C	80°C



Legend		
Change due to valve replacement	Valve replacement leak tests	Failure leak rate 23 ml/min (0.00138 m ³ /hr)

Summary

Thin PTFE tape (white) with compound passed with no leakage. Spikes measured in 8302 were leaks from the stem of valve 1.

Sealant Suitability

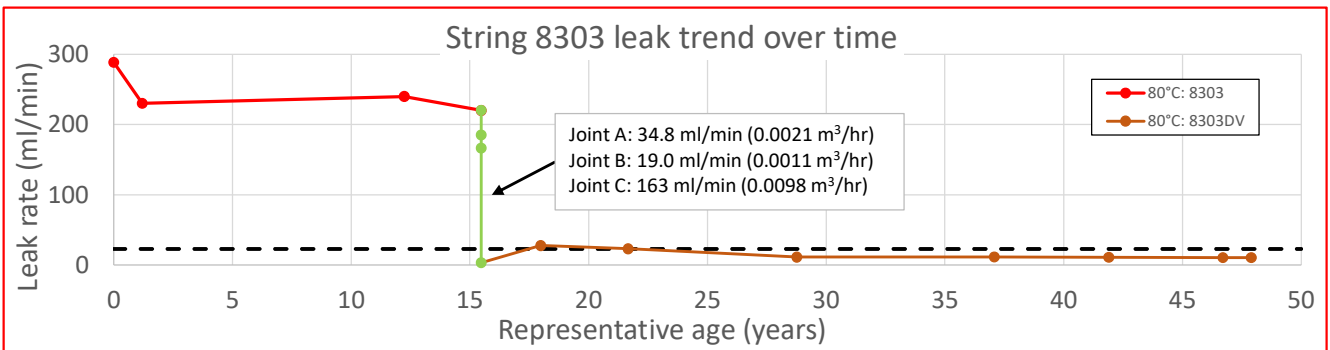
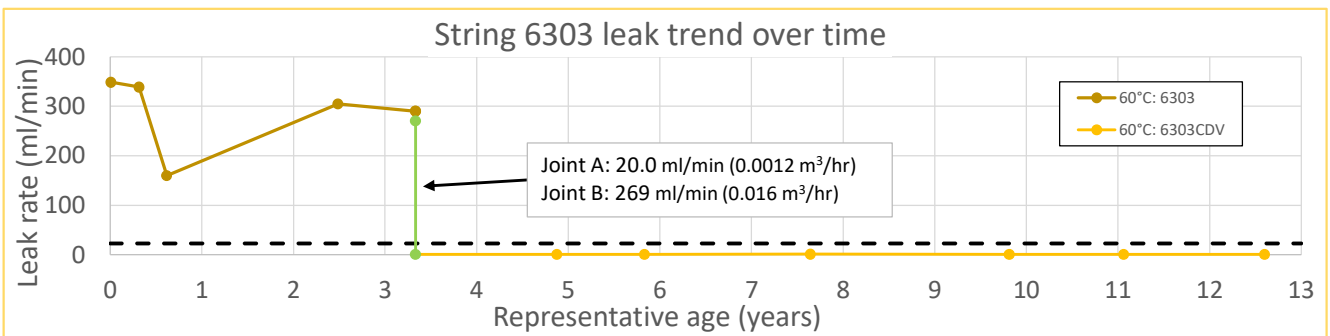
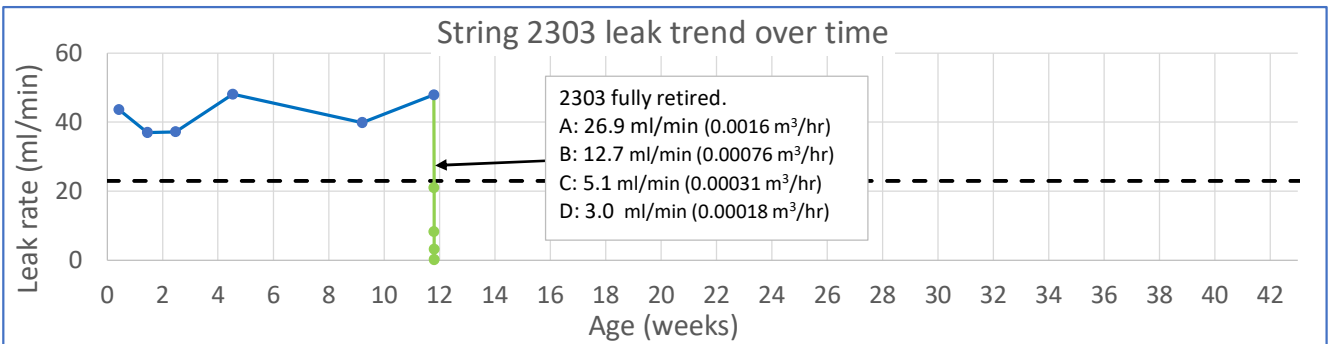


Sealant 303

Unsintered PTFE Tape

String reference		
2303	6303	8303

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Retirement
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

All three thin PTFE tape (white) strings leaked from their initial assembly. Attempts to remake leak-free strings were not successful. Leakage did not increase over time.

Due to air ingress and potential invalidation of experimentation, 2303 was fully retired, 2 joints in 6303 and 3 joints in 8303 were sealed and retired.

Sealant Suitability

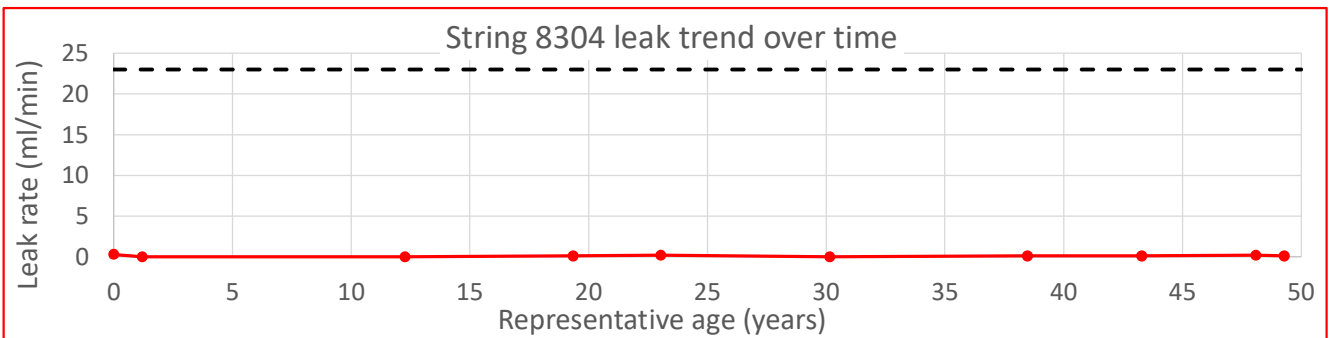
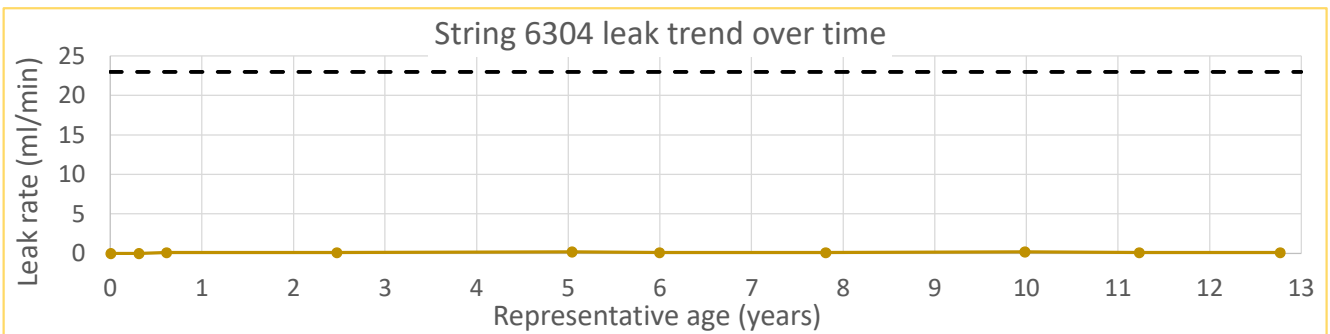
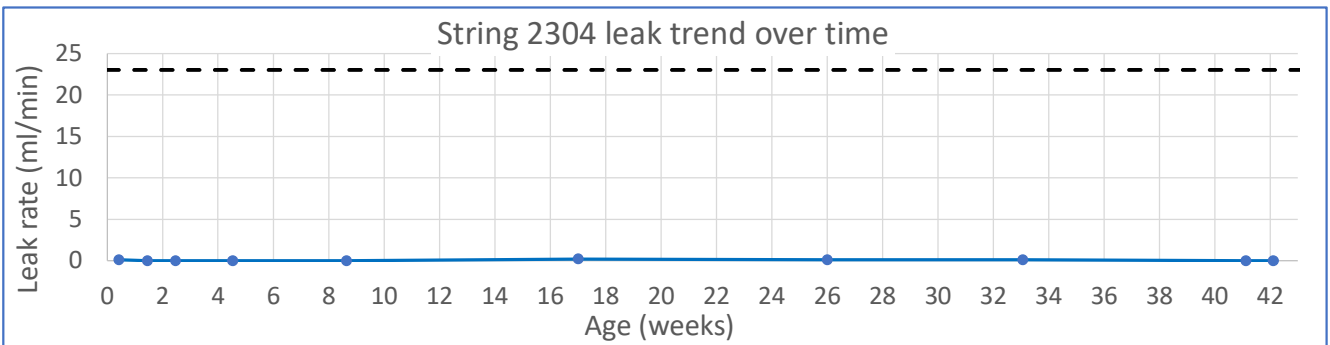


Sealant 304

Unsintered PTFE Tape & Non-hardening jointing compound

String reference		
2304	6304	8304

Temperature of storage		
20°C	60°C	80°C



Legend

- Change due to valve replacement
- Valve replacement leak tests
- Failure leak rate 23 ml/min (0.00138 m³/hr)

Summary

Thick PTFE tape (yellow) with compound passed with no leakage.