TECHNICAL PAPER

API 6A/ISO 10423 Appendix F.1.13.5.2 Elastomer Immersion Tests - what do I need to know?

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API 6A/ISO 10423 Appendix F.1.13.5.2 Elastomer Immersion Tests are common benchmarks for elastomeric sealing materials being used in the oil and gas industry. Phrases like Approved to API6A ISO10423 'Sour', Tested to ISO 10423 (API 6A) Sour Gas and Certified to API6A Material Class FF/HH are commonplace on material datasheets and the promotional material of elastomeric seal vendors. However, those phrases alone give very little indication of a material's pedigree for service in critical applications. Without understanding the specific testing undertaken it is impossible to assess the suitability of the material in the intended service environment.

This document summarises the test requirements of the API 6A, Annex F and ISO10423 Elastomer Immersion Tests and highlights areas to be considered when interpreting results.

API Specification 6A Wellhead and Tree Equipment is now in its 21st edition and is a modified national adoption of **ISO 10423**, 4th Edition: Petroleum and Natural Gas Industries - Drilling and Production Equipment - Wellhead and Christmas Tree Equipment. Thus, API 6A and ISO 10423 are often referred to together or interchangeably.

API 6A/ISO 10423 Appendix F.1.13.5.2 provides a standardised method of evaluating the thermochemical performance of sealing materials for service in liquids and gasses representative of the service environment. This is done by comparing the physical and mechanical properties of the sealing material prior to and following exposure to standard test fluids, temperatures and pressures.

Figure 1 — Elastomer seals tested to ISO 10423 (API 6A)

For elastomeric materials, the standard test requires the aging of ASTM D412 dumbbell test pieces in the liquid phase of the test chamber, at the specified temperature, at a pressure of 6.9MPa (1,000psi) for a minimum duration of 160 hours. The percentage change in measured properties are then reported. Test samples are also visually inspected for damage such as cracking, blistering, dissolution etc. The specifications do, however, encourage bespoke media, temperature, pressure and duration to be utilised when this is more representative of the intended service environment.

The specifications state that Appendix F.1.13.5.2 alone must not be used to validate the suitability of a product and an appropriate pressure and temperature test shall also be carried out.

Test Fluid

The standard test fluids are listed in Table 1. The test fluids comprise of a hydrocarbon liquid phase over-pressurised with a gas mixture. The hydrocarbon liquid phase in which the test pieces are immersed comprise 60% of the test vessel volume plus 5% de-ionised water. The remaining 35% of the vessel volume is made up of the gas phase.

Material Class		Gas Phase	Hydrocarbon Phase
AA / BB	General Service – Low CO_2	5% CO_2 / 95% CH_4	At manufacturer's discretion
CC	General Service – High CO_2	80% CO_2 / 20% CH_4	
DD/EE	Sour Service – Low CO_2	10% H_2S / 5% CO_2 / 85% CH_4	
FF/HH	Sour Service – High CO_2	10% H_2S / 80% CO_2 / 10% CH_4	

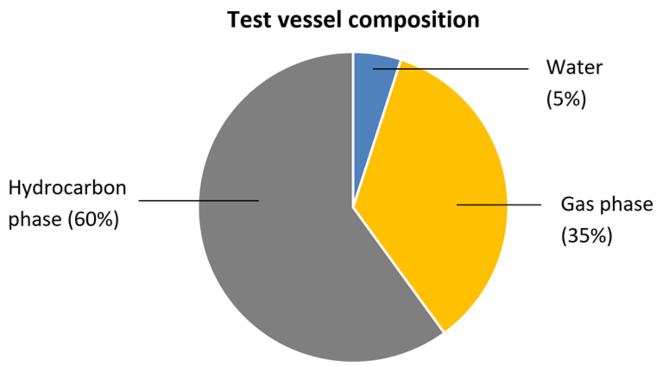
Table 1 — API 6A/ISO 10432 standard test fluids for non-metallic seals

The hydrocarbon liquid phase is chosen at the manufacturer's discretion but will most commonly be kerosene or the 70/20/10 heptane/cyclohexane/toluene cocktail used in ISO 23936-2:2011 (NORSOK M-710 Edition 3) sour gas testing.

Depending on the material class, the gas phase has different proportions of CO_2 and CH_4 plus, where sour service is required, H_2S.

The choice of material class should be that which is most representative of the intended service environment. While there is general acceptance that sealing materials that are suitable in FF/HH will also be suitable in DD/EE etc, caution should be employed when selecting materials on that basis. Further, as the hydrocarbon phase is not fully defined this means that for some applications a benign fluid is chosen such that interaction with test gas is the primary focus, alternatively application conditions might dictate that an aggressive hydrocarbon phase is required.

Hence two sets of testing that might both be naively referred to as FF/HH testing or DD/EE testing may produce two different outcomes. When making a comparison between materials it is imperative that this is considered.



Temperature

The standard test temperatures are listed in Table 2 on the following page. The specifications state that the test temperature should be either:

- The maximum temperature rating for the appropriate temperature classification for the equipment being tested (e.g. Material for a Class K would be tested at +82°C/+180°F)

OR

- The maximum temperature at the seal location as established by product testing or design analysis.

For elastomeric sealing materials, testing is typically carried out at +121°C/+250°F for NBR compounds (in accordance with Classes U and V) and +177°C/+350°F for FKMs and FEPMs (in accordance with Class X).

<table>
<tr><th>Temperature Classification</th><th>Operating Range °C</th><th>Operating Range °F</th></tr>
<tr><td>K</td><td>-60 to +82</td><td>-75 to +180</td></tr>
<tr><td>L</td><td>-46 to +82</td><td>-50 to +180</td></tr>
<tr><td>N</td><td>-46 to +60</td><td>-50 to +140</td></tr>
<tr><td>P</td><td>-29 to +82</td><td>-20 to +180</td></tr>
<tr><td>R</td><td colspan="2">Room Temperature</td></tr>
<tr><td>S</td><td>-18 to +66</td><td>0 to +150</td></tr>
<tr><td>T</td><td>-18 to +82</td><td>0 to +180</td></tr>
<tr><td>U</td><td>-18 to +121</td><td>0 to +250</td></tr>
<tr><td>V</td><td>+2 to +121</td><td>+35 to +250</td></tr>
<tr><td>X</td><td>-18 to +177</td><td>0 to +350</td></tr>
<tr><td>Y</td><td>-18 to +343</td><td>0 to +650</td></tr>
</table>

Table 2 — API 6A/ISO 10432 temperature ratings

HNBR compounds are typically tested at +149°C/+300°F although this doesn't coincide with any of the standard temperature classifications – the temperature chosen to reflect applications where it has been determined, as per the above, that thermal performance above that of NBR is required.

Test temperatures may also vary slightly depending on the geographical region where testing is carried out (typically, Fahrenheit is used in North America and Celsius in Europe). It is common to see test temperatures of +120°C, +150°C or +175°C where the temperature in Celsius has been rounded off from the Fahrenheit equivalent. In these cases, it is normally acceptable to use the results interchangeably with +121°C (250°F), +149°C (300°F) or +177°C (350°F) respectively.

Pass/fail criteria

The procedure outlined in API 6A/ISO 10432 Appendix F.1.13.5.2 is not intended to be used as a pass/fail test but rather to allow for the evaluation of how a seal is likely to perform in the intended service environment and to allow a comparison of the performance of different materials under standard test conditions. The specifications therefore do not provide guidance about acceptable levels of change in the measured properties of the materials being tested. The reasons for this are clear; in some applications given changes in hardness and tensile strength (for example) may be acceptable but in others the same level of change could have catastrophic results. Consideration as to how representative (and how aggressive) the test fluid is to the service environment must also be considered when this is assessed.

<table>
<tr><th>Property</th><th>Allowable Change</th></tr>
<tr><td>Volume</td><td>-5/+25%</td></tr>
<tr><td>Hardness (IRHD)</td><td>+10/-20</td></tr>
<tr><td>Tensile Strength</td><td rowspan="3">±50%</td></tr>
<tr><td>Modulus</td></tr>
<tr><td>Elongation at Break</td></tr>
</table>

Table 3 — NORSOK M-710 Acceptance Criteria

As a reference, especially where API 6A/ISO 10432 Appendix F.1.13.5.2 is being carried out as a benchmarking exercise rather than for a specific application, the acceptance criteria defined in ISO 23936-2:2011 (NORSOK M-710 Edition 3) "*Qualification of non-metallic sealing materials and manufacturers*", are often employed as shown in Table 3.

How many elastomers do PPE have tested to API 6A/ISO 10432 Appendix F.1.13.5.2?

PPE has had numerous elastomer compounds tested by third party accredited laboratories to this standard, including various HNBR, FEPM, FKM and FFKM materials. To allow a direct comparison of materials, testing has been carried out using the FF/HH gas and at temperatures that are commensurate with the material type.

PPE Grade	Material Type	Test Temperature	Hydrocarbon phase
A85H	FEPM	175°C / 350°F	70/20/10 heptane/cyclohexane/toluene
A90H	FEPM	175°C / 350°F	70/20/10 heptane/cyclohexane/toluene
G75B	FFKM	200°C / 392°F	70/20/10 heptane/cyclohexane/toluene
G75LT	FFKM	200°C / 392°F	70/20/10 heptane/cyclohexane/toluene
G75M	FFKM	200°C / 392°F	70/20/10 heptane/cyclohexane/toluene
G90LT	FFKM	177°C / 350°F	Kerosene
G92E	FFKM	177°C / 350°F	Kerosene
V74C	FKM	177°C / 350°F	Kerosene
V75J	FKM	175°C / 350°F	70/20/10 heptane/cyclohexane/toluene
V75T	FKM	175°C / 350°F	70/20/10 heptane/cyclohexane/toluene
V76F	FKM	177°C / 350°F	Kerosene
V91A	FKM	177°C / 350°F	Kerosene
V91J	FKM	177°C / 350°F	Kerosene
V91K	FKM	177°C / 350°F	Kerosene
Z70B	HNBR	150°C / 300°F	70/20/10 heptane/cyclohexane/toluene
Z83B	HNBR	150°C / 300°F	70/20/10 heptane/cyclohexane/toluene
Z85B	HNBR	150°C / 300°F	70/20/10 heptane/cyclohexane/toluene
Z85L	HNBR	150°C / 300°F	70/20/10 heptane/cyclohexane/toluene
Z95X	HNBR	150°C / 300°F	70/20/10 heptane/cyclohexane/toluene

Table 4 — PPE material grades tested to ISO10423 (API 6A)

Summary

In summary, when assessing the suitability of elastomeric sealing material for a specific service environment it is important to remember that materials certified to API 6A/ ISO 10432 Appendix F.1.13.5.2 may not have been tested using the same test regime. It is important to understand whether the material class, temperature and hydrocarbon phase used are indicative of the intended service environment.

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