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SOUR FLUID IMMERSION TESTING OF N90BR ACCORDING TO API 6A

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1. SYNOPSIS

Sour fluid immersion testing N90BR according to API 6A Annex. F (ISO 10423:2009, section F.1.13.5.2) at 100 °C for 160 hours has been completed. Performance was evaluated by measuring changes in mass, volume and tensile property levels at room temperature.

Changes in mechanical and physical properties have been measured after immersion in the hydrocarbon oil phase of a multi-phase sour fluid containing 10_{mol}% hydrogen sulphide (10/80/10 $_{mol}$% $H_2S/CO_2/CH_4$).

Material performance is summarized in the grids below.

Material Physical Performance after Immersion Test

Test Material	PROPERTY CHANGE		
	Mass (%)	Volume (%)	Hardness (Shore A, Units)
N90BR	2.95	6.79	-9

Material Tensile Performance after Immersion Test

Test Material	PROPERTY CHANGE			
	M50 (%)	M100 (%)	Tensile strength (%)	Elongation at break (%)
N90BR	-44.9	-28.3	-16.2	-12.6

N90BR was intact after the immersion test, with no signs of blistering or degradation.

2. OBJECTIVES

MCM SpA have contracted Element Hitchin to carry out testing on N90BR according to API 6A Annex F (ISO 10423:2009 “Petroleum and natural gas industries – Drilling and production equipment – Wellhead and Christmas tree equipment” section F.1.13.5.2 – Immersion testing of materials for sour service).

This document summarises the work undertaken by Element Hitchin.

3. METHOD

3.1 Material

The N90BR was delivered to Element Hitchin on 27th June 2017 in the form of 200 mm x 200 mm x 2 mm slabs and logged in with a unique reference number according to Element quality procedure (Table 3.1).

Table 3.1: Material

Test Material	Element Quality Reference Number	Type of Material
N90BR	M21486	FKM

From supplied sheet, ISO 37 Type 2 dumbbells were cut using a certified cutter.

The sour gas mixture was procured from Compressed Gas Solutions Ltd.; the certificate of composition is shown in Appendix A.

3.2 Test conditions

The test conditions are summarised in Table 3.2.

Table 3.2: API 6A (ISO 10423 – F.1.13.5.2) – test conditions

Temperature	100 °C
Pressure	69 barg (1,000 psi)
Gas phase (35%)	10/80/10 $_{mol}$% $H_2S/CO_2/CH_4$ [class FF/HH]
Liquid phases (65%)	60% vessel volume 70/20/10 heptane/cyclohexane/toluene 5% vessel volume water
Tensile replicates	5
Exposure time	160 hours, minimum

The samples were exposed in the hydrocarbon liquid phase. Changes in physical and mechanical properties (tensile and swelling) of the materials were measured at room temperature before and after the immersion test.

3.3 Test procedure

The immersion test was undertaken in a pressure vessel (rated to 15,000 psi) equipped with an external band heater, calibrated pressure transducer, internal thermocouple (located among the test specimens) and an isolation needle valve (Figure 3.1). Cell pressure and temperature were logged continuously by a PC running dedicated data acquisition software.

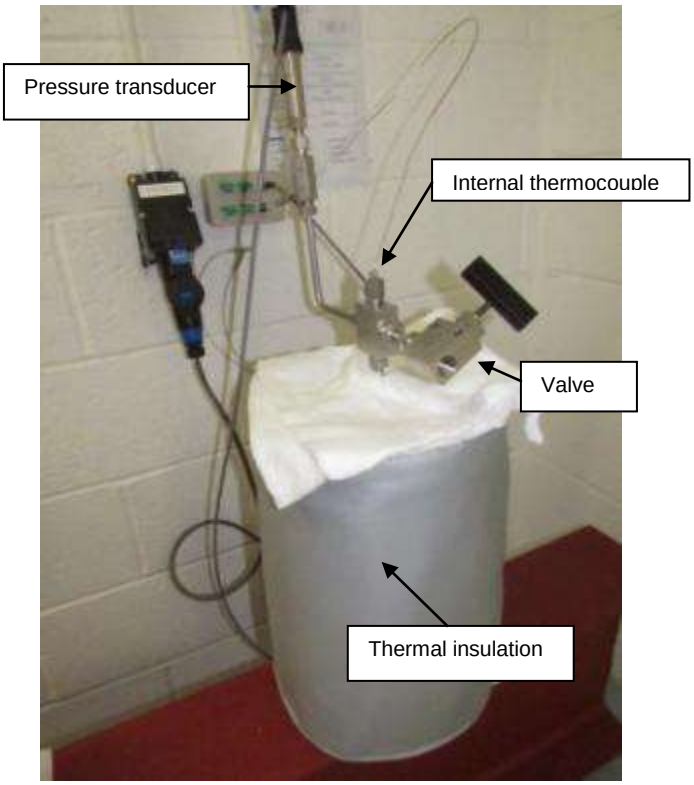


Figure 3.1: Pressure vessel for sour fluid test equipped with pressure sensor, internal thermocouple and needle valve.

5 replicates of the material were placed into the pressure vessel, onto which the required volume of test liquids was added. The materials were completely submerged. The cell was flushed with nitrogen to remove residual air (oxygen). The cell was heated to test temperature and then 10/80/10 $_{mol}$% $H_2S/CO_2/CH_4$ gas mixture added to 70 bar.

Appropriate temperature and pressure alarm limits were set on the pressure vessel. The test pressure and temperature logs are plotted together in Appendix B. The pressure inside the vessel decreases throughout the test. This drop in pressure is due to the absorption of the gas into the liquid phase. There is no leak in the test apparatus. Thus, the pressure was boosted on 3 separate occasions. After 160 hours of immersion, the test vessel was cooled naturally to room temperature, which led to a pressure decrease. The samples remained in the vessel under these conditions before the gas was released at less than 1 bar/minute into a chemical scrubber.

The samples were left in the pressure vessel undisturbed before being retrieved. Within 30 minutes, the specimens were weighed. A calibrated milligramme electronic balance was used for weighing.

Tensile testing employed a Zwick Z050 screw-driven test machine equipped with a calibrated 5 kN load cell and contacting arm extensometer for strain measurement of tensile dumbbells. Test speed was 500 mm/minute.

4. RESULTS

API 6A (ISO 10423 – F.1.13.5.2) does not provide guidelines as to what levels of change are acceptable for the different properties measured. Table 4.1 shows the changes in mass and volume measured after immersion for 160 hours at 100 °C. The quoted values are the mean of three

samples. The tensile measurements are the mean of five samples. The values in brackets are the percentage changes for tensile properties.

Table 4.1: Changes in mass, volume and tensile properties of N90BR after immersion testing in hydrocarbon liquid phase

Material		Mass change (%)	Volume change (%)	Hardness (Shore A, Units)	Modulus at 50% elongation (MPa)	Modulus at 100% elongation (MPa)	Tensile strength (MPa)	Elongation at break (%)
N90BR	Unaged	-	-	91	5.20	8.24	19.0	303
	Aged	2.95	6.79	83	2.86 (-45%)	5.91 (-28%)	15.9 (-16%)	265 (-13%)

No damage or blistering on any of the samples was evident after the immersion test; all samples were intact after immersion test. Swelling was due to fluid absorption at test temperature.

5. CONCLUSIONS

Sour fluid immersion testing was performed on N90BR according to API 6A (ISO 10423 – F.1.13.5.2) at 100 °C for minimum 160 hours. Samples were immersed in the hydrocarbon oil phase of a multi-phase sour fluid containing 10% hydrogen sulphide (10/80/10 $_{mol}$% $H_2S/CO_2/CH_4$).

Property Performance after Immersion Test

Test Material	PROPERTY CHANGE						
	Mass (%)	Volume (%)	Hardness (Shore A, Units)	Modulus at 50% elongation (%)	Modulus at 100% elongation (%)	Tensile strength (%)	Elongation at break (%)
N90BR	2.95	6.79	-9	-44.9	-28.3	-16.2	-12.6

6. APPENDIX A. GAS CERTIFICATE

ELEMENT HITCHIN

Compressed Gas Solutions Ltd

CYLINDER TYPE	50L STEEL	DATE	15/05/2017
VALVE TYPE	BS15	CGS REF	2017-334
STABILITY	15/05/2019	CUSTOMER REF	EHI014808PO–1
CERTIFIED BY	PL		
PRESSURE	32 BAR		
VOLUME	1.97 M3		
NET WEIGHT	3.3 KGS		

CYLINDER NO.

13710688

CERTIFICATE OF COMPOSITION

COMPONENT	REQUESTED VALUE	CERTIFIED VALUE
HYDROGEN SULPHIDE 2.5	10.0%	9.99%
METHANE 4.5	10.0%	10.0%
CARBON DIOXIDE 4.5	BALANCE	BALANCE

ALL UNITS ARE MOLAR, WITH A MIXTURE ACCURACY OF ±2%

KEEP THE MIXTURE ABOVE 0°C TO PREVENT CONDENSATION OF THE CONDENSABLE PRODUCTS, IF PRESENT.

PRODUCTS ARE FILLED GRAVIMETRICALLY AND TRACEABLE TO STANDARDS 218M CALIBRATED AT THE NATIONAL PHYSICAL LABORATORY, TEDDINGTON.

Compressed Gas Solutions Ltd, J Reid Trading Estate, Factory Road, Sandycroft, Flintshire, CH5 2QJ, +44 (0) 1244 520688

Figure 6.1: Test gas certificate of composition

7. APPENDIX B. PRESSURE AND TEMPERATURE PLOTS

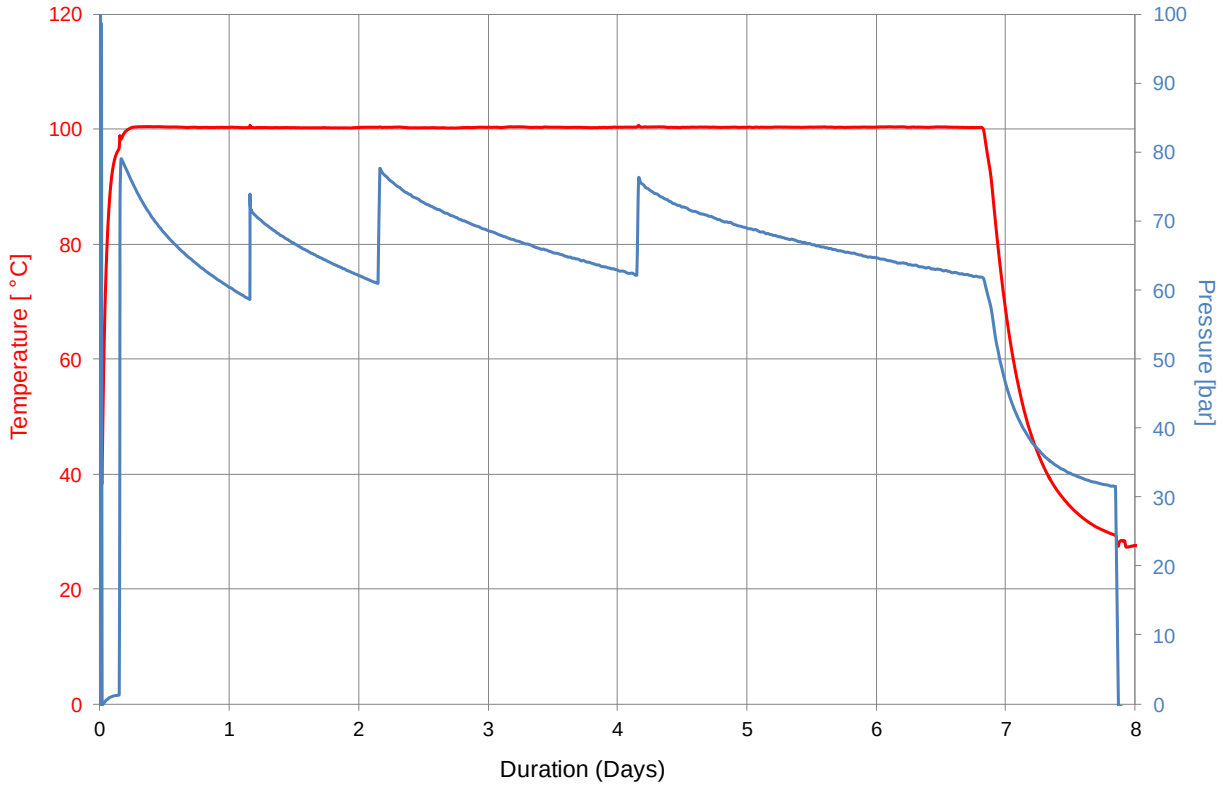


Figure 7.1: Pressure/temperature plots for the sour immersion test