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SOUR FLUID IMMERSION OF KIFK 11 FKM AED -18°C ELASTOMERIC MATERIAL ACCORDING TO NACE TM0187-2011

Version Control

Revision	Date	Description of Change + Reason for Change	Prepared by	Reviewed by
Initial issue	06-May-2021	First issue	K.Somani	M.Lewan

TABLE OF CONTENTS

1. SYNOPSIS

Sour fluid immersion testing of KIFK 11 FKM AED -18°C elastomeric material according to NACE TM0187-2011 at 175 °C for 160 hours has been completed. Performance was evaluated by measuring changes in mass, volume, hardness, compression set and tensile property levels at room temperature.

Changes in mechanical and physical properties have been measured after immersion in the gaseous phase of a multi-phase sour fluid containing 20$_{mol}$% hydrogen sulphide (20/5/75 $_{mol}$% $H_2S/CO_2/CH_4$).

Material performance is summarized in the grids below.

Material Physical Performance after Immersion Test

COMPOUND	PROPERTY CHANGE			
	Mass Change (%)	Volume Change (%)	Hardness Change (Shore A units)	Compression set (%)
KIFK 11 FKM AED -18°C	1.47	2.71	-1	36

Material Tensile Performance after Immersion Test

COMPOUND	PROPERTY CHANGE			
	50% modulus (%)	100% modulus (%)	Tensile strength (%)	Elongation at break (%)
KIFK 11 FKM AED -18°C	16	22	-15	-33

There were no signs of chemical ageing of KIFK 11 FKM AED -18°C after the test. Tensile and mass/volume/hardness (MVH) samples of KIFK 11 FKM AED -18°C were intact. Acceptance criteria are not provided in the NACE TM0187 standard. For interest, if the widely used criteria given in the ISO 23936-2 standard are applied, then KIFK 11 FKM AED -18°C are considered acceptable.

2. OBJECTIVES

Industrial Spares Manufacturing & Trading Co. (ISMAT) have contracted Element Hitchin to carry out testing on their elastomeric material - KIFK 11 FKM AED -18°C according to NACE TM0187-2011. Test conditions described in the NACE TM0187 standard and Element's internal procedure 14.21 are applicable. The test fluid is multi-phase with the testpieces located in the gas, the major phase by volume. The test gas contains 20% hydrogen sulphide (H_2S) and the exposure temperature is 176 °C (350 °F). The exposure duration is 160 hours, minimum. Material performance will be evaluated by measuring changes in mass, volume, and tensile property levels, all measured at room temperature.

This document summarises the work undertaken by Element Hitchin.

3. METHOD

ISMAT delivered elastomeric compound KIFK 11 FKM AED -18°C to Element Hitchin on 13th January 2021 in the form of 150 x 150 x 2 mm sheets and compression set buttons. Samples were logged in with a unique reference number according to Element quality procedure (Table 3.1).

Table 3.1: Test materials

COMPOUND	Element Quality Reference Number	Samples delivered
KIFK 11 FKM AED -18°C	M25619 and M25620	3 off Buttons and 2 off Sheets

A total of 10 tensile dumbbells of ISO 37 type 2 were stamped from these sheets by Element using a certified cutter; the level of replication was five for the fluid exposure. Also, three replicate rectangles of size 5 cm × 2.5 cm were cut for mass/volume/hardness (MVH) measurements. Samples before exposure are shown in Appendix A.

The sour gas mixture was procured from Compressed Gas solutions Ltd, the certificate of composition is shown in Appendix B.

3.1 Test conditions

The test conditions are summarised in Table 3.2.

Table 3.2: NACE TM0187 – test conditions

Temperature	175 °C (±3 °C)
Pressure	69 barg (1,000 psi) at ambient temperature
Gas phase (90%)	20/5/75 $_{mol}$% $H_2S/CO_2/CH_4$
Aqueous phase (5%)	Water
Oil phase (5%)	50% n-decane, 25% n-hexane, 20% n-octane, 5% toluene
Tensile replicates	5
Exposure time	160 hours, minimum

KIFK 11 FKM AED -18°C samples were exposed in the gas phase. Changes in physical and mechanical properties (tensile and swelling) of the material were measured at room temperature before and after the immersion test.

3.2 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.

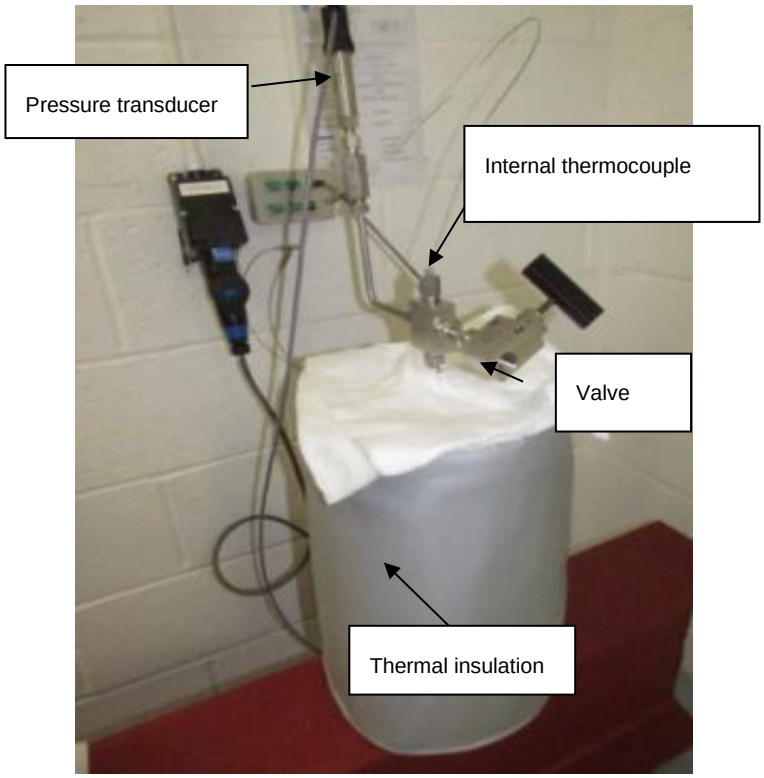


Figure 3.1: Pressure vessel for sour fluid test equipped with pressure sensor, internal thermocouple and needle valve.

5 replicates of the tensile dumbbells, 3 replicates of the mass-volume-hardness (MVH) rectangles, and 3 replicates of the compression set buttons KIFK 11 FKM AED -18°C were exposed together. The tensile dumbbells and MVH specimens of each compound were marked to aid identification. The buttons were placed inside compression fixtures (2 parallel circular metal plates with a metal spacer in-between) and compressed by 25%.

The liquids were added to the pressure vessel, and the samples placed on a tripod so that none of the samples came into contact with the liquid. The cell was flushed with nitrogen to remove residual air (oxygen). 20/5/75 $_{mol}$% $H_2S/CO_2/CH_4$ gas mixture was added to 1000 psi (ca.70 bar). The vessel was then heated to test temperature (175°C) which raised the pressure to 1709 psi (118 bar).

Appropriate temperature and pressure alarm limits were set on the pressure vessel. After 160 hours at 176°C the gas was released into a chemical scrubber at 1 bar/minute using a dedicated blowdown rig, under computer control. The vessel was then cooled naturally overnight. After flushing with nitrogen the cell was opened and the samples were retrieved and tested immediately; mass in air and water, hardness and tensile measurements. The samples in the compression jigs were removed and placed on a poor thermally conducting surface for 30 minutes at ambient temperature before re-measuring the thickness.

A calibrated milligramme electronic balance was used for weighing. Hardness was measured using a calibrated Shore A hardness tester. Hardness was measured at three locations and the average reported.

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

Figure 4.1 shows the pressure/temperature plot for the sour fluid immersion test. Figure 4.2 shows the decompression profile at test temperature. The test was run between 26th January 2021 and 4th February 2021.

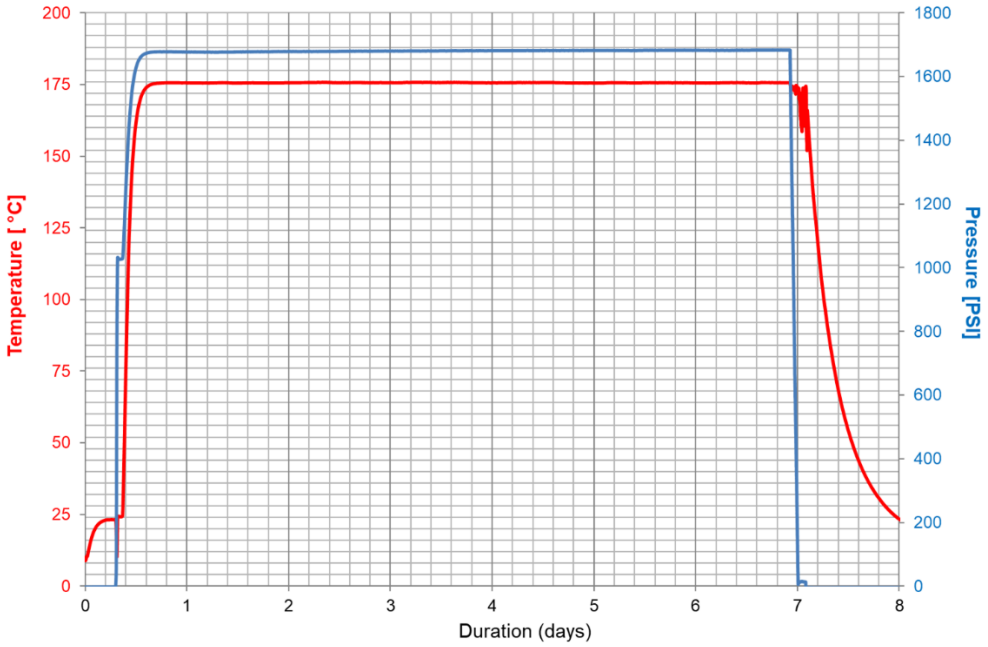


Figure 4.1: Temperature and pressure vs. time for immersion testing

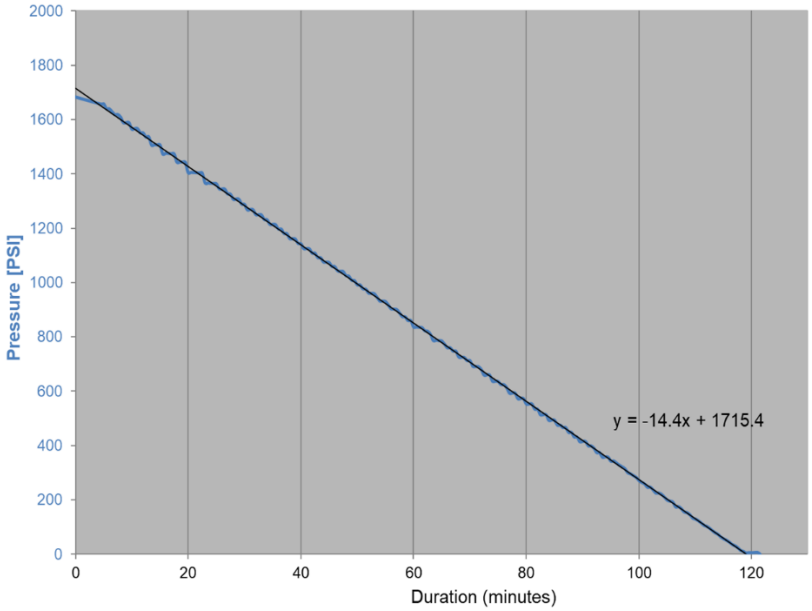


Figure 4.2: Decompression profile after immersion period

Photographs of KIFK 11 FKM AED -18°C after test are shown in Appendix C.

Table 4.1 lists the changes in mass, volume and hardness measured for KIFK 11 FKM AED -18°C after 160 hours at 175 °C.

Table 4.1: Mass, volume and hardness change of elastomers after immersion testing

Compound	Element Material Reference	Replication	After immersion			Mean change		
			Mass Change (%)	Volume change (%)	Hardness Shore A Units	Mass Change (%)	Volume change (%)	Hardness Shore A Units
KIFK 11 FKM AED -18°C	M25620	1	1.50	2.57	-1	1.47	2.71	-1
		2	1.47	3.02	0			
		3	1.44	2.55	-1			

The average hardness of the as-received elastomeric compound KIFK 11 FKM AED -18°C was 86 Shore A units. Liquid vapour absorption is responsible for the volume expansion and hardness reduction of 1 unit was due to minor swelling. There were no signs of stiffening after the exposure test.

The average compression set level for the three replicate buttons is listed in Table 4.2. Minor surface cracks were found on all elastomers buttons after immersion.

Table 4.2: Average compression set of elastomers

Compound	Element Material Reference	Replication	Compression Set (%)	Mean Compression Set (%)
KIFK 11 FKM AED -18°C	M25621	1	37.84	36
		2	34.92	
		3	35.95	

The tensile data before and after testing is summarised in Table 4.3.

Table 4.3: Tensile properties of Elastomers after the test

Material ID	Condition	Modulus at 50% strain	% Change	Modulus at 100% strain	% Change	Tensile strength at Break	% Change	Elongation at Break	% Change
		MPa		MPa		MPa		%	
KIFK 11 FKM AED -18°C	Unaged	5.60	16	10.23	22	16.46	-15	177	-33
	luid Exposed	6.50		12.45		14.06		118	

There was no visual change to KIFK 11 FKM AED -18°C after fluid exposure; all specimens were intact. Tensile performance was moderately affected. Although the H_2S level in the test gas mixture is high (20%), Element expectation is that the fluoroelastomer compound will not significantly age within a week at 175 °C.

5. CONCLUSIONS

After the exposure, no visible degradation or damage was apparent on KIFK 11 FKM AED -18°C (tensile dumbbells, MVH specimens and compression set buttons).

Elastomeric performance is summarized in Table 5.1.

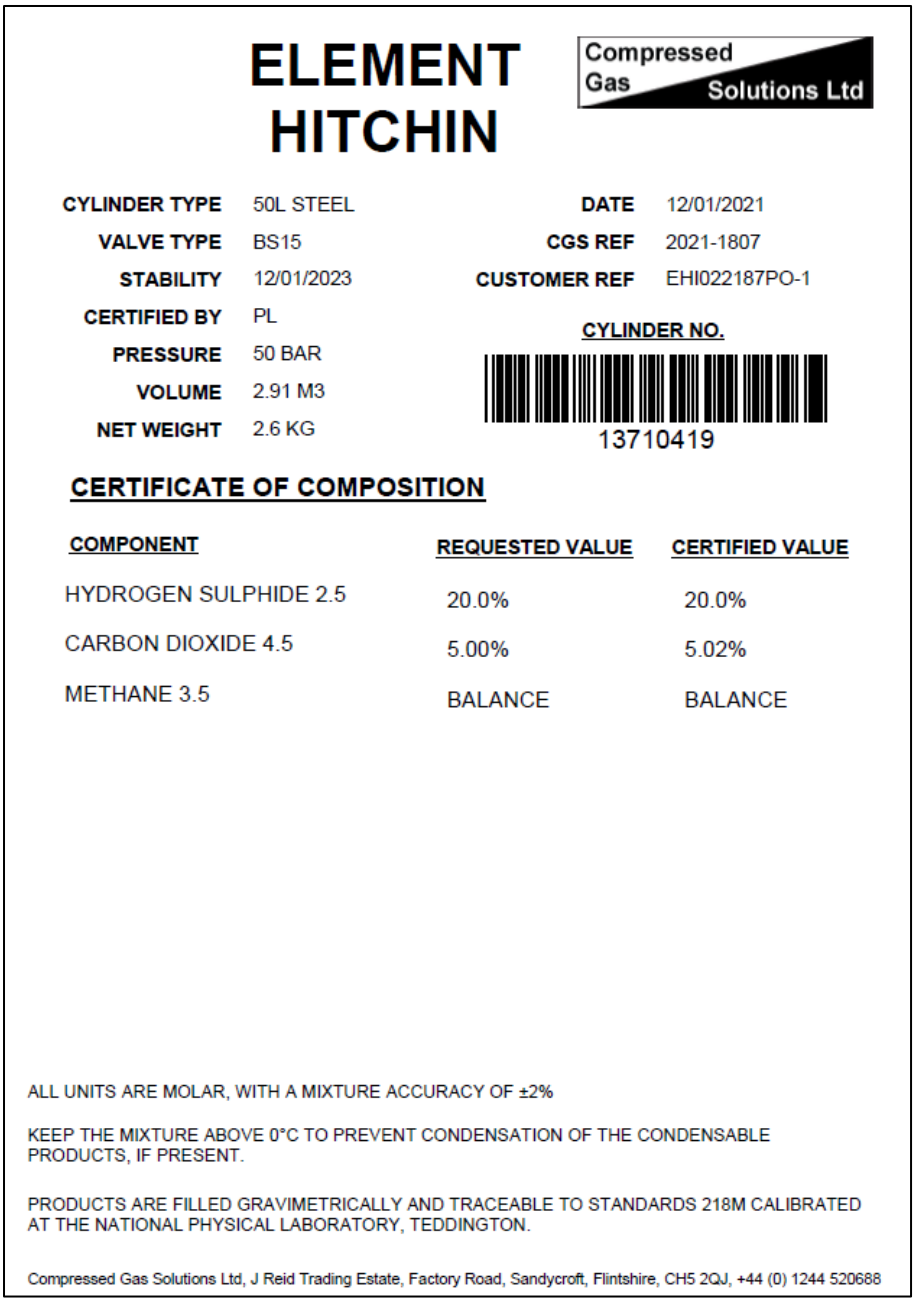
Table 5.1: Elastomer performance summary

PROPERTY	KIFK 11 FKM AED -18°C
Visual	No change
Mass uptake (%)	1.47
Volume swell (%)	2.71
Hardness change (Shore A units)	-1
50% Modulus change (%)	16
100% Modulus, change (%)	22
Tensile strength change (%)	-15
Elongation at break change (%)	-33
Compression set (%)	36

APPENDIX A As-received KIFK 11 FKM AED -18°C and before immersion

Figure A.1 As received material/before start of immersion: KIFK 11 FKM AED -18°C

APPENDIX B Gas certificate



ELEMENT HITCHIN

Compressed Gas Solutions Ltd

CYLINDER TYPE	50L STEEL	**DATE**	12/01/2021
VALVE TYPE	BS15	**CGS REF**	2021-1807
STABILITY	12/01/2023	**CUSTOMER REF**	EHI022187PO-1
CERTIFIED BY	PL		
PRESSURE	50 BAR		
VOLUME	2.91 M3		
NET WEIGHT	2.6 KG		

CYLINDER NO.

13710419

CERTIFICATE OF COMPOSITION

COMPONENT	REQUESTED VALUE	CERTIFIED VALUE
HYDROGEN SULPHIDE 2.5	20.0%	20.0%
CARBON DIOXIDE 4.5	5.00%	5.02%
METHANE 3.5	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 B.1 Test Gas Certificate

APPENDIX C Photograph of samples after immersion

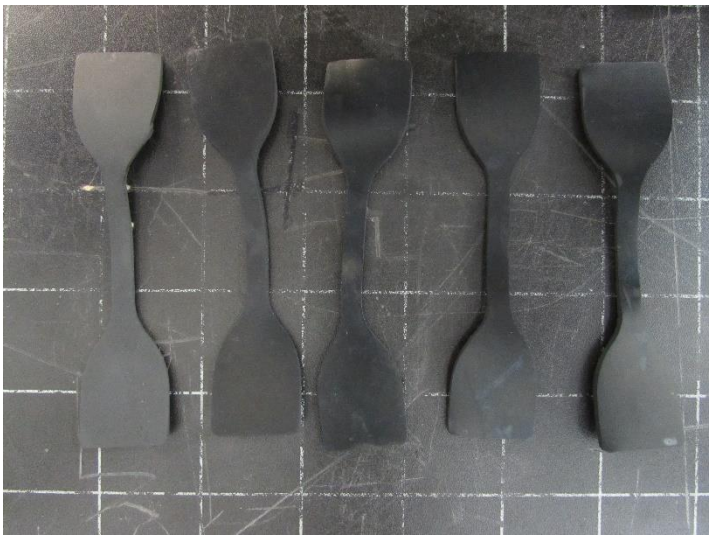


Figure C.1: **Tensile dumbbells of - KIFK 11 FKM AED -18°C after immersion**

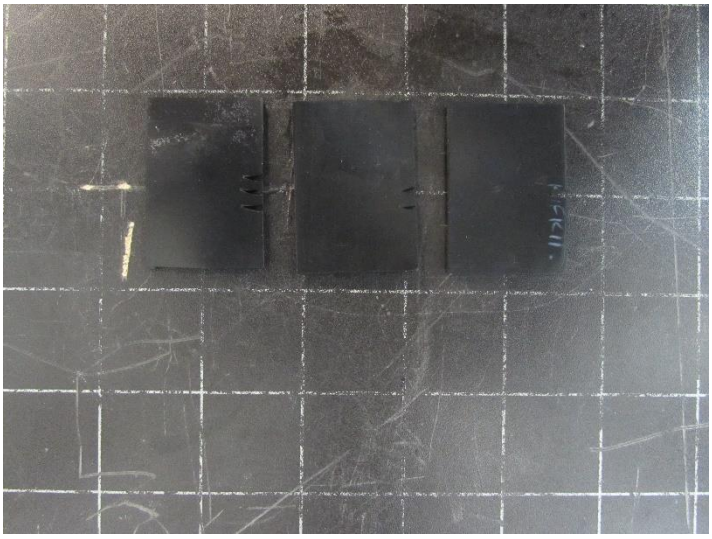


Figure C.2: **MVH specimens of - KIFK 11 FKM AED -18°C, after immersion**

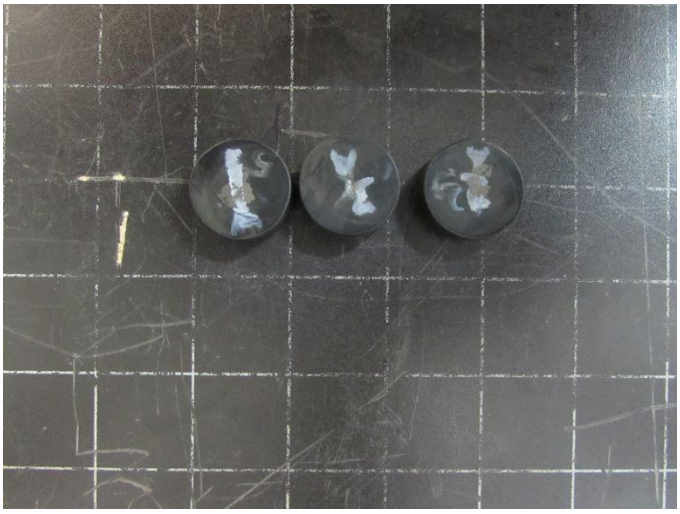


Figure C.3: **Compression set button specimens of - KIFK 11 FKM AED -18°C, after immersion**