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Date: 27th April 2017
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Report Number: C4339/N9036
Client Reference: PO 2016-28307

EVALUATION OF N9036 ACCORDING TO ISO 23936-2 / NORSOK M-710, EDITION 3

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1. SYNOPSIS

The sour fluid resistance of N9036 has been investigated according to the ISO 23936-2 / NORSOK M-710 standard, Edition 3. Exposure testing was conducted at 95, 110 and 125 °C, for up to 56 days; the test gas mixture contained 10% H_2S. Material performance was evaluated by tracking changes in mass, volume, hardness and tensile property levels, all recorded at room temperature. Material performance is summarised in the grid below. All changes are based upon the as-received material properties.

MCM Material Identification	Material Type	SWELL RANGE [%]	Hardness change [Units]	MAXIMUM % CHANGE			
				M50	M100	Tensile strength	Elongation at break
N9036	FKM	18 - 19	-14	-22	-2.8	-15	-19

Observations:

- There are no signs of deterioration; all specimens were retrieved intact after sour fluid exposure.
- Tensile performance is insensitive to fluid exposure temperature once swelling is discounted, indicating the absence of a chemical ageing process.
- The general low swell and high tensile property level retention are in line with expectations.

The absence of change attributable to chemical ageing means that N9036 can be described as having, according to the standard, "indefinite life at the lowest test temperature and temperatures below this".

2. OBJECTIVES

MCM Spa required Element Hitchin to carry out immersion tests with eight elastomeric materials in accordance with the NORSOK M-710 standard, Edition 3. Tensile specimens were used to quantify changes of room temperature mechanical and physical performance as a function of exposure time and temperature.

In order to accelerate chemical ageing (if it occurs) to obtain results in reasonable time schedules, test temperatures need to be well above design temperatures but not so high that unrepresentative reactions might occur. In practice, the boundary is not known; hence it is best to select sensible temperatures for immersion tests. Three exposure test temperatures are typically chosen to enable extrapolation of results back to (lower) service temperatures, following the Arrhenius principle.

Tensile dumbbells were exposed to a standard NORSOK M-710 fluid mixture containing 10 mol% H_2S in the test gas mixture. Samples were periodically removed for testing at room temperature.

3. METHOD

Since part of the NORSOK standard M-710 concerns test procedures for the determination of change, or rate of change, of relevant physical properties, at least three exposure times at each temperature are necessary for the application of the Arrhenius model. The exposure test conditions specified by MCM are listed in Table 3.1. 60 bar is applied at 30 °C. The test fluid composition chosen by MCM and the phase distribution is shown in Table 3.2.

Table 3.1: Exposure test conditions

TEMPERATURE (°C)	SAMPLING INTERVALS (days)	PRESSURE (bar)
95	7, 14, 28, 56	60 bar at 30 °C
110		
125		

Table 3.2: Test Fluid

VESSEL VOLUME (%)	FLUID COMPOSITION
30	10% H_2S, 5% CO_2, 85% CH_4*
10	Deionised water (conductivity < 5 mS)
60	70% heptane, 20% cyclohexane, 10% toluene

* Appendix A shows the test gas certificate from the supplier CK Gas Products Ltd. (UK).

Test material details are given in Table 3.3. Each material was given a unique Element number, for traceability according to the ISO 9001 quality system.

Table 3.3: Material details

MCM Material Identification	Element Quality Reference Number	Material	Samples delivered
N9036	M21074	FKM	Tensile dumbbells: ISO 37 Type 2 Compression buttons (29.0 mm diameter and 12.5 mm thick) Rectangles (Mass, volume, hardness measurements)

Replicate tensile specimens were placed in appropriate pressure vessels. Each cell was equipped with an external band heater, calibrated pressure transducer, internal thermocouple (located among the test samples) and isolation needle valve. Cell pressure and temperature were logged continuously by a PC running dedicated data acquisition software: the pressure and temperature logs are given in Appendix B.

The same quantity of test gas (60 bar) was added to each vessel for each exposure interval. The addition temperature was 30 °C (Table 3.1).

At each sampling interval, the vessels were cooled overnight to 30 – 40 °C, before slowly venting residual gas pressure into a chemical scrubber. From each cell, five tensile testpieces and the rectangles were removed. Weighing in air and water was carried out immediately. Where possible, tensile testing was carried out within 2 hours, but if this was not possible the samples were stored in fresh NORSOK oil at room temperature until testing could be undertaken.

All tensile testing was performed at room temperature using a Zwick Z250 test machine equipped with a calibrated 5 kN load cell. The test rate was 500 mm/minute. Properties reported include modulus, tensile strength and elongation at break.

Weighing employed a calibrated milligramme electronic balance.

The following changes in properties are deemed acceptable for elastomers (Table 3.4) according to the NORSOK standard.

Table 3.4: NORSOK acceptance criteria for elastomers

PROPERTY	CRITERION RANGE
Volume change	+25/-5%
Modulus, tensile strength, elongation at break	+/-50%
Hardness change	+10/-20 units (+5/-20 units when initial hardness is 90)

4. RESULTS

The material performance is described in the sub-section below. The baseline for the mechanical property change is that of as-received material.

4.1 N9036 [FKM]

The average levels of mass, volume, hardness and tensile properties as a function of fluid exposure time and temperature for this material are listed in Table 4.1. Graphical representations of the data are shown in Appendix C.

Table 4.1: N9036. Mass, volume, hardness and tensile performance

Temp.	Exposure period (days)	Mass change (%)	Volume change (%)	Hardness (Shore A)	Modulus @50% strain (MPa)	Modulus @100% strain (MPa)	Tensile strength (MPa)	Elongation at Break (%)
95°C	0	-	-	92	6.0	9.9	13.4	146
	2	7.3	17.4	79	4.8	9.1	10.9	123
	7	8.0	18.3	81	3.7	6.8	11.2	177
	14	7.5	16.7	78	5.1	10.1	11.6	117
	28	7.4	17.0	77	5.0	10.1	12.0	120
	56	8.0	18.5	81	5.1	10.1	11.9	120
110°C	0	-	-	92	6.0	9.9	13.4	146
	2	8.1	18.7	80	3.5	6.2	11.0	189
	7	8.0	18.5	79	4.8	9.4	11.0	119
	14	7.2	16.4	76	4.7	9.4	11.6	125
	28	7.1	16.1	77	4.8	9.6	11.8	126
	56	8.1	18.7	79	4.7	9.6	11.7	123
125°C	0	-	-	92	6.0	9.9	13.4	146
	2	8.5	19.0	80	4.4	8.7	10.9	129
	7	8.1	18.4	78	4.3	8.7	11.1	130
	14	7.7	16.8	76	4.6	9.2	11.4	126
	28	7.4	16.4	75	4.5	9.3	11.1	121
	56	8.6	19.1	78	4.7	9.7	11.4	118

4.1.1 Mass, volume and hardness changes

Equilibrium swell is between 18.5 - 19.1%, which is well within the upper acceptance limit of ISO 23936-2 (+25%).

At all three temperatures there is an initial drop in hardness followed by stabilisation, with final values being 9 - 14 units less than that of as-received material. This is within the lower acceptance limit of ISO 23936-2 of -20 units.

4.1.2 Tensile property level changes

Modulus and tensile strength both show a decrease after the initial exposures, whilst elongation at break increases. This is the expected response to liquid absorption. This is a physicochemical effect and not an ageing phenomenon.

Further exposure results in an increase in modulus and tensile strength, and a decrease in elongation at break which all stabilise after 2 weeks exposure. Generally Arrhenius plots for material life predictions cannot be derived because the changes in properties are not temperature dependent; that is, rate of change does not increase with increasing exposure temperature – there is no uniform long-term indication of change between test temperatures.

As a check, linear regression analysis (Figure 4.1) has been performed with the data.

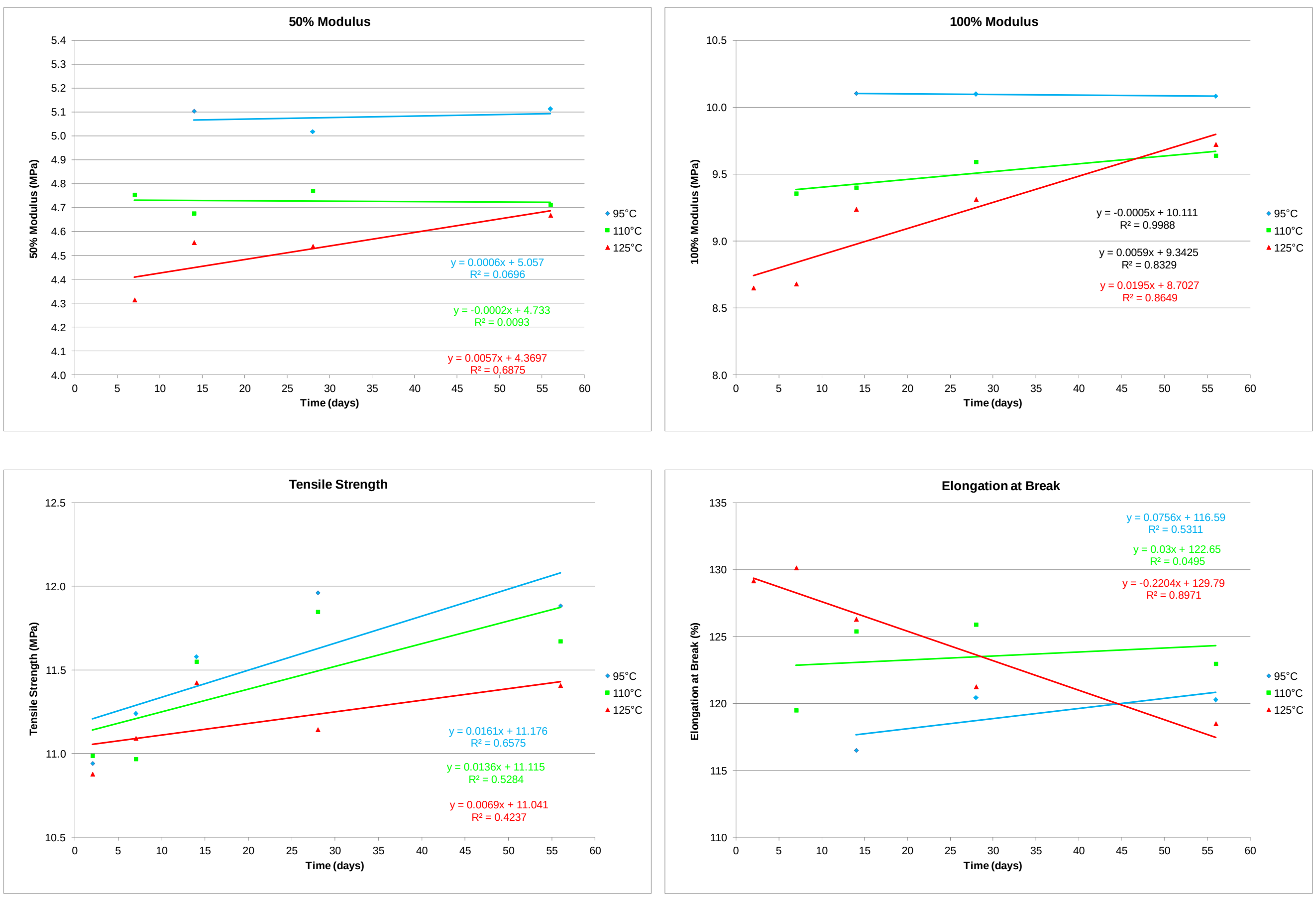

Figure 4.1: N9036. Linear regression lines for tensile properties.

The times to reach a 50% change in each property are listed in Table 4.2; these were calculated by applying the equation for the best fit straight line through each data set.

The table highlights the erratic behaviour of all the data. The rate of change does not increase with increasing exposure temperature.

After 56 days exposure the changes are small with the largest change being 22%.

Table 4.2: Times for property levels to change by 50% at each temperature

Property	Temperature (°C)	Time to Change 50% (days)	
		Decrease	Increase
50% Modulus	95	-	4322
	110	11790	-
	125	-	368
100% Modulus	95	10122	-
	110	-	796
	125	-	219
Tensile strength	95	-	325
	110	-	395
	125	-	765
Elongation at break	95	-	769
	110	-	1886
	125	296	-

5. SUMMARY AND CONCLUSIONS

The material performance is summarized in Table 5.1, which lists the swell and percent change in tensile property levels at the end of the three exposure tests, and described further below the table.

Table 5.1: Material performance summary

MATERIAL	EXPOSURE TEMP [°C]	SWELL [%]	Hardness (units)	% CHANGE			
				M50	M100	Tensile strength	Elongation at break
N9036	95	18.5	81	-15	+1.7	-12	-18
	110	18.7	79	-22	-2.8	-13	-16
	125	19.1	78	-22	-2.0	-15	-19

- There are no visible signs of deterioration; all specimens were retrieved intact.
- All swelling is less than 25%; acceptable according to the standard.
- Room temperature tensile property level change is always within ±50% of the as-received level.
- Tensile performance is insensitive to fluid exposure temperature, indicating the absence of a chemical ageing process.

The absence of change attributable to chemical ageing means that N9036 can be described as having, according to the standard, “indefinite life at the lowest test temperature and temperatures below this”.

APPENDIX A [TEST GAS CERTIFICATE]

CK SPECIAL GASES LTD

Ashby Suite, Wellington House
Leicester Road, Ibstock
Leicestershire, LE67 6HP
Tel: 01530 267209
Fax: 01530 382110

Certificate of Composition

Customer No.	EH1013412PO-1	**Cylinder No.**	10/26404
Our Ref	6245	**Cylinder Valve**	BS15
Nett Wt (kg)	4.8 KG	**Cylinder Size**	50L STEEL

Component	Requested Value	Certified Value
HYDROGEN SULPHIDE 2.5	10.0%	9.98%
CARBON DIOXIDE 4.5	5.00%	5.01%
METHANE 3.5	BALANCE	BALANCE

All units are MOLAR, with a mixture accuracy of ±2%.

Products are filled gravimetrically and traceable to standards 218M calibrated at the National Physical Laboratory, Teddington.

Pressure	93 bar	**Volume**	5.9 M3	**Valid Until**	19/10/2018
Certified by	PL	**UN No.**	1953	**Date**	19/10/2016

Figure A.1: Test gas mixture certificate of composition.

APPENDIX B [PRESSURE – TEMPERATURE PLOTS]

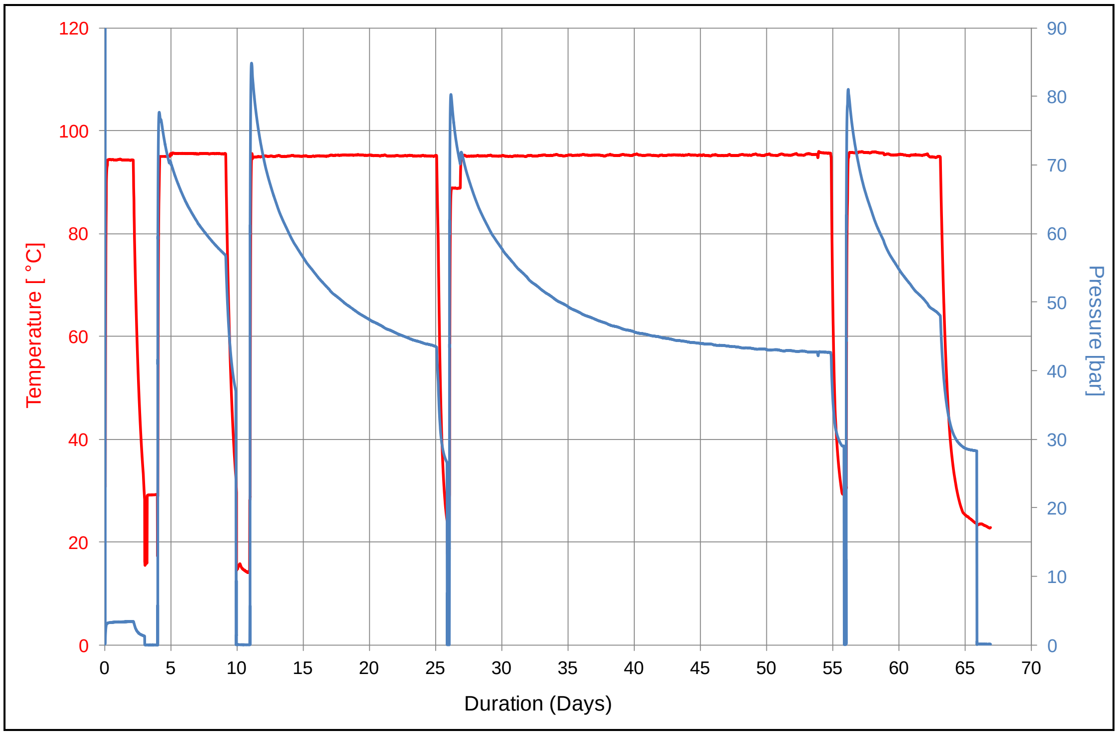

Figure B.1: Temperature and pressure vs. time for exposure test at 95 °C.

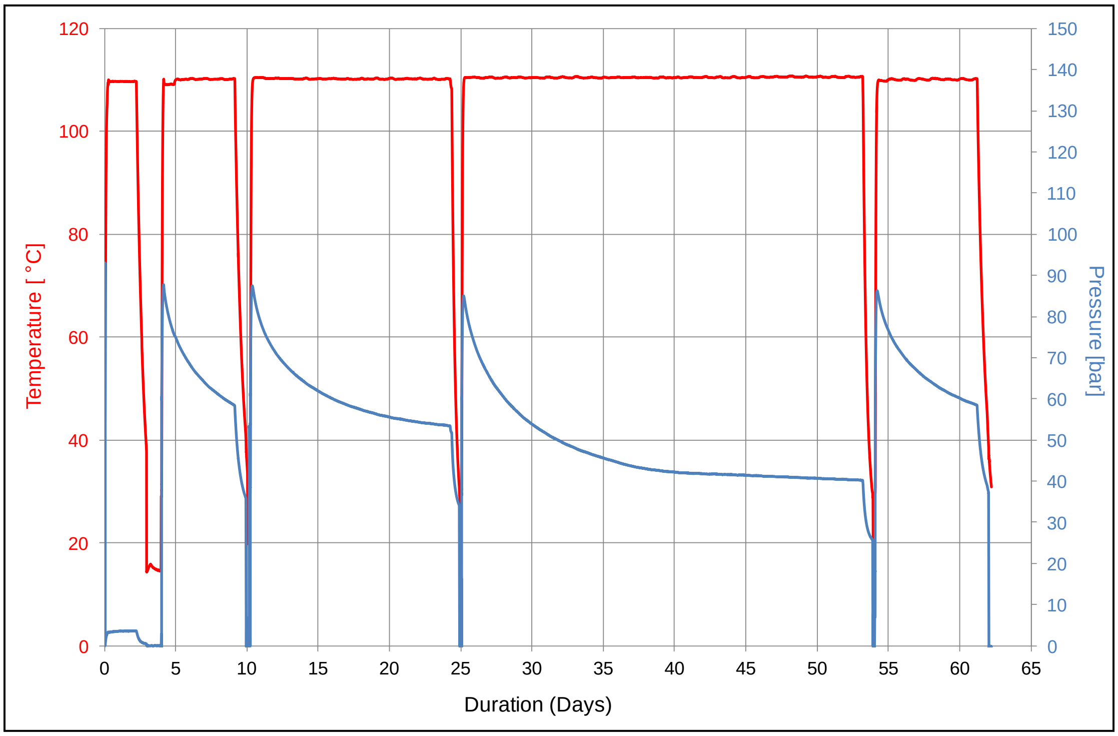

Figure B.2: Temperature and pressure vs. time for exposure test at 110 °C.

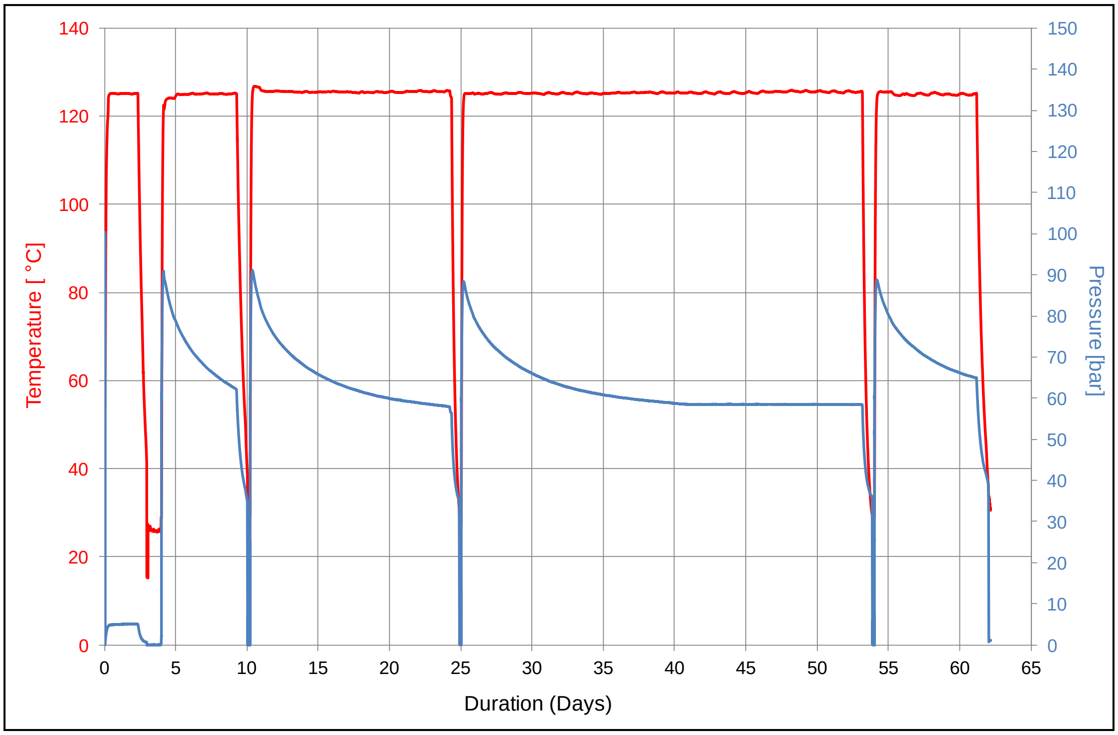

Figure B.3: Temperature and pressure vs. time for exposure test at 125 °C.

APPENDIX C [N9036 PERFORMANCE CHARTS]

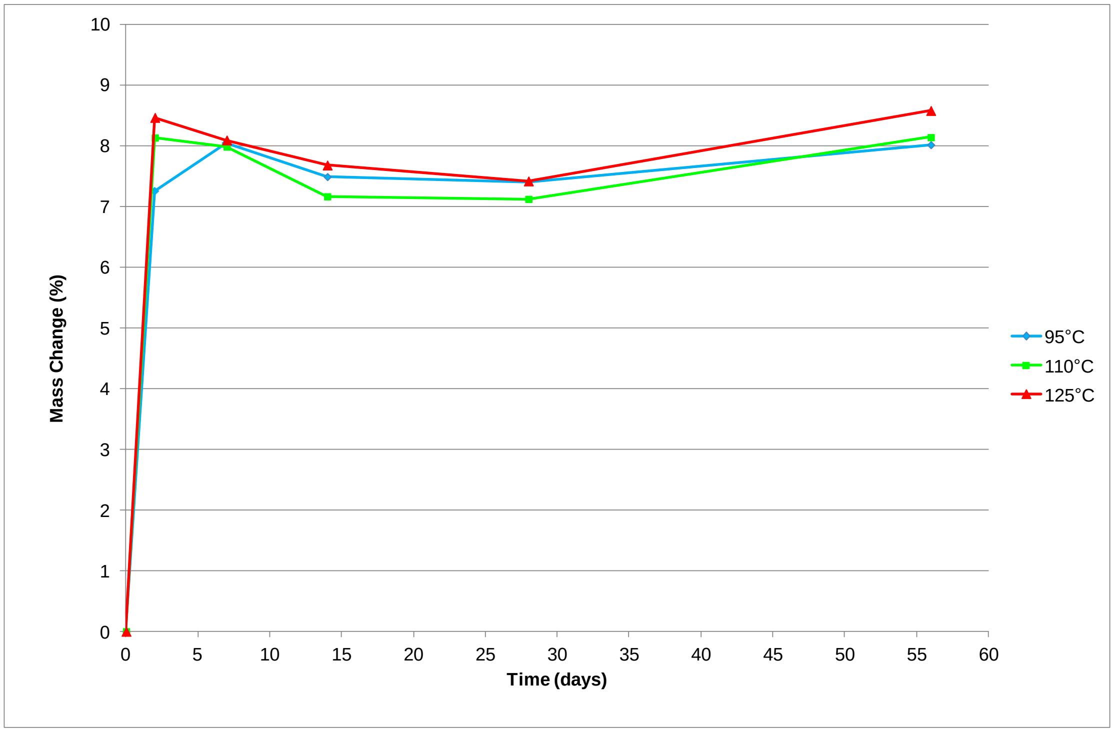

Figure C.1: N9036. Mass change vs. fluid exposure time and temperature.

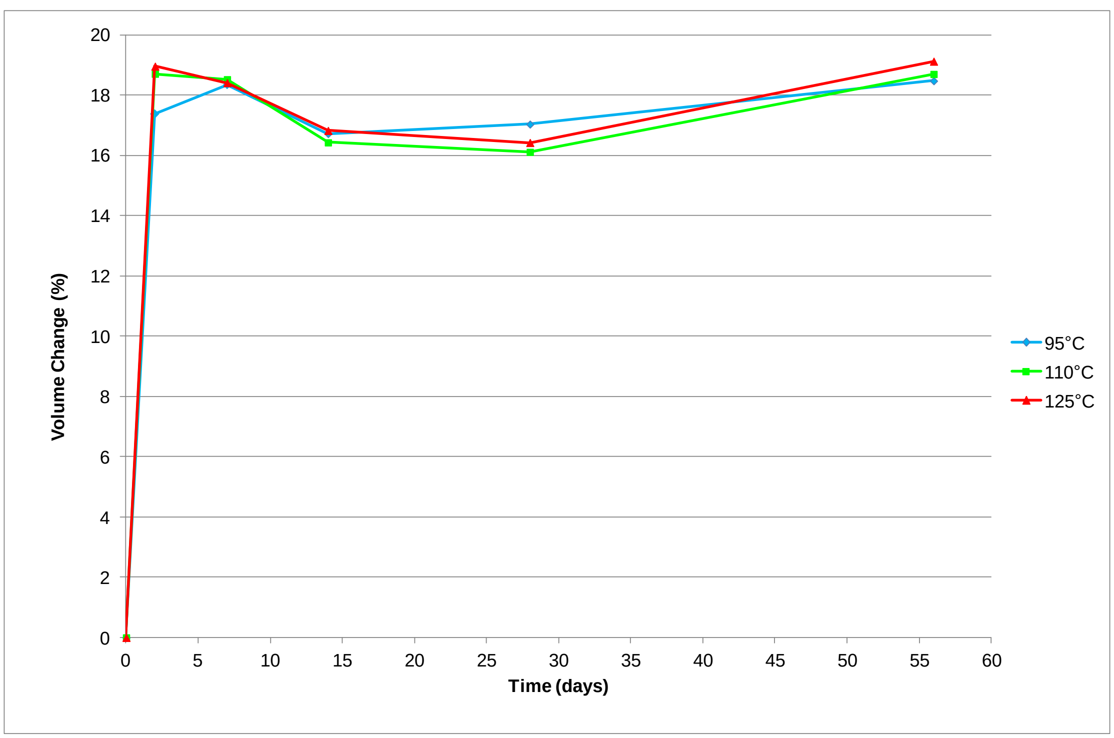

Figure C.2: N9036. Volume change vs. fluid exposure time and temperature.

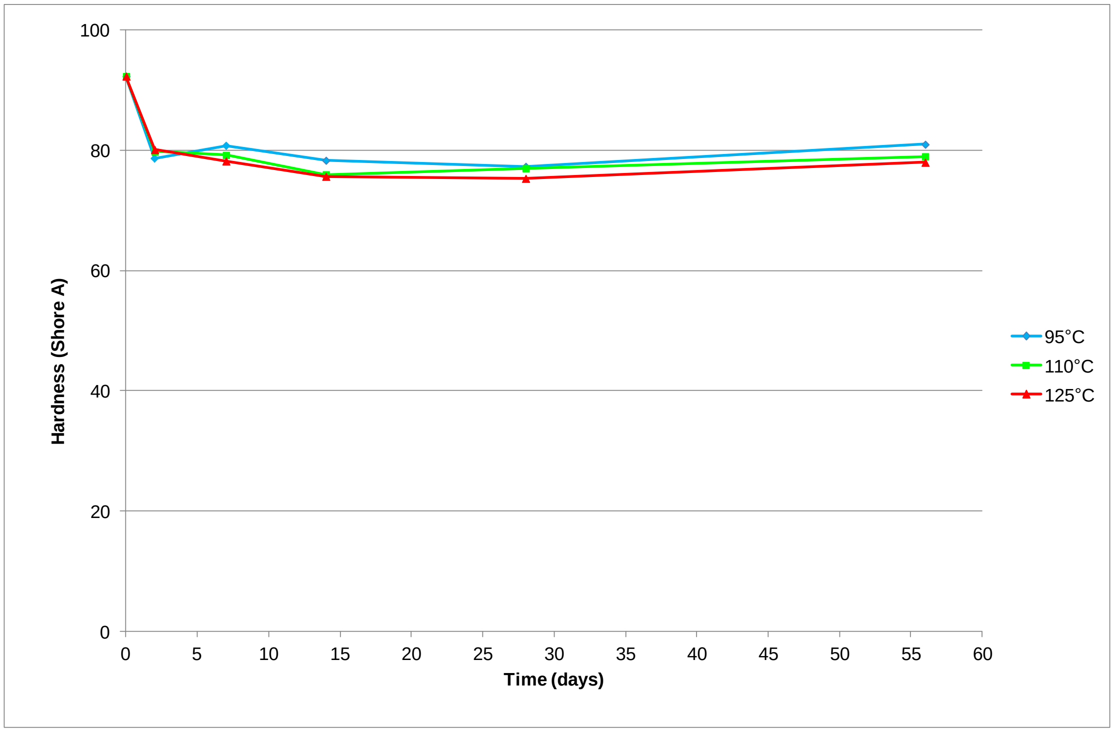

Figure C.3: N9036. Average hardness vs. fluid exposure time and temperature.

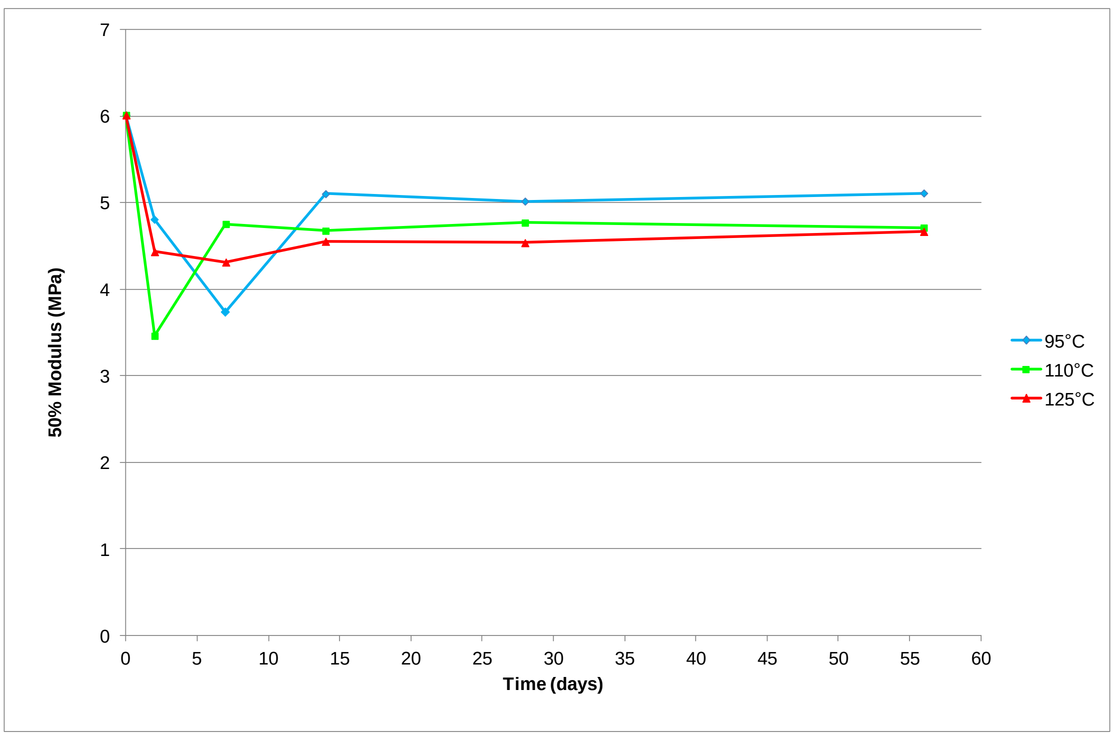

Figure C.4: N9036. Average M50 vs. fluid exposure time and temperature.

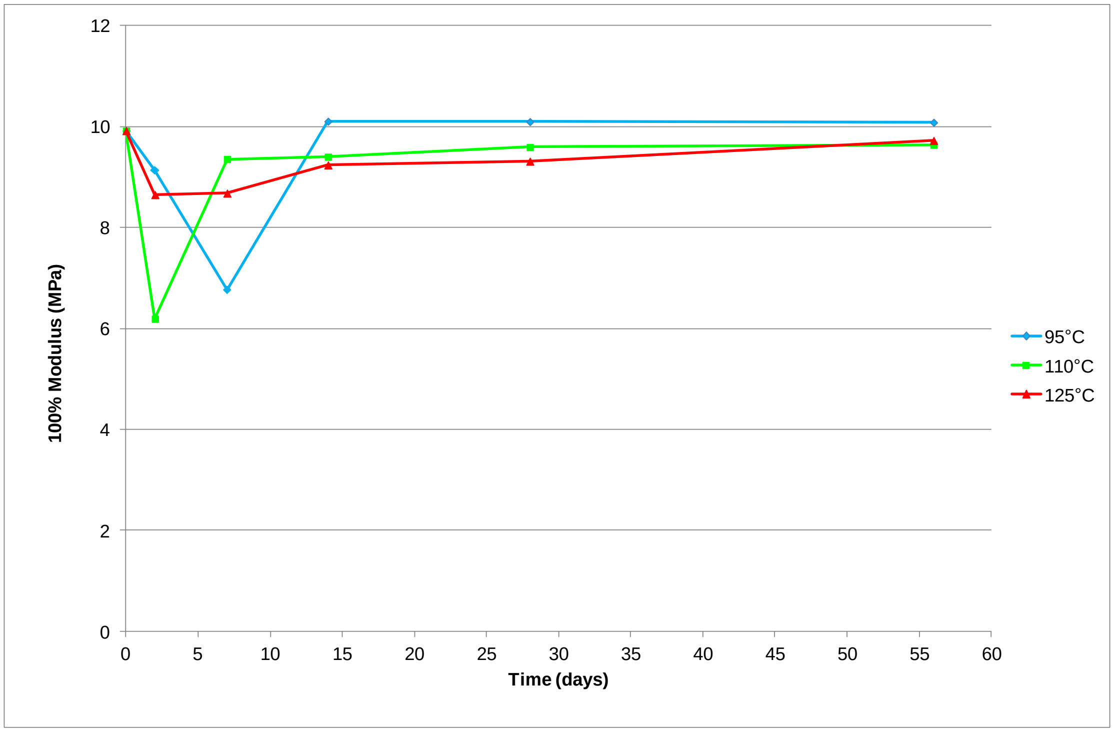

Figure C.5: N9036. Average M100 vs. fluid exposure time and temperature.

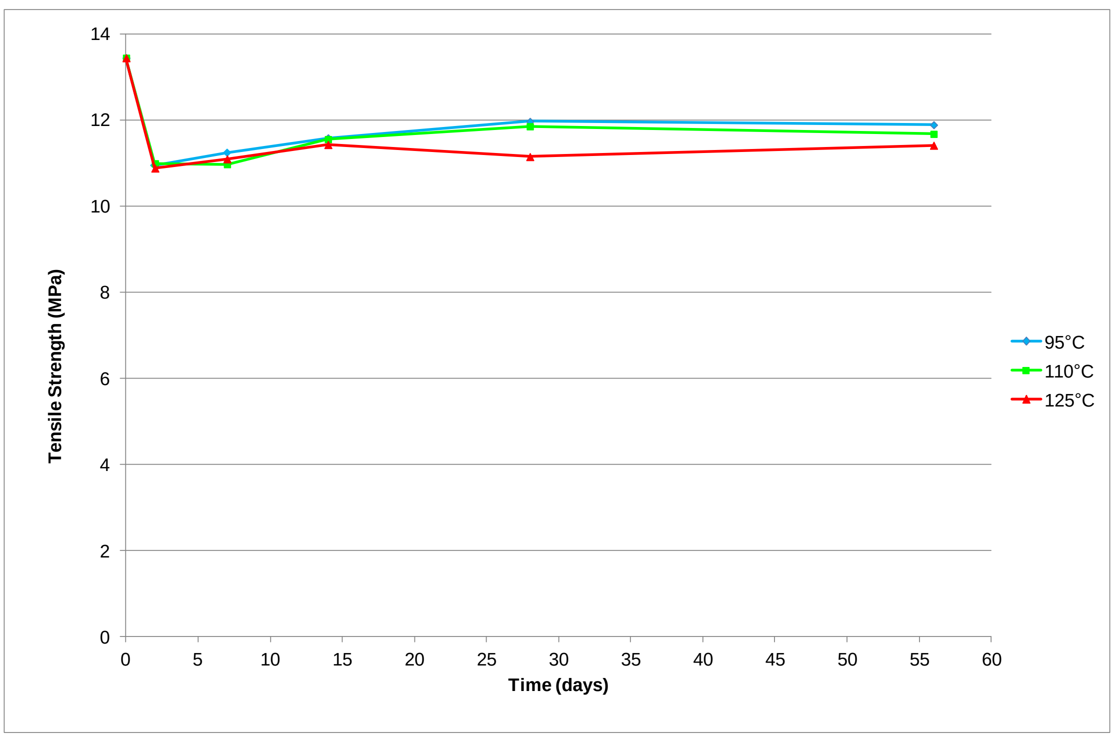

Figure C.6: N9036. Average tensile strength vs. fluid exposure time and temperature.

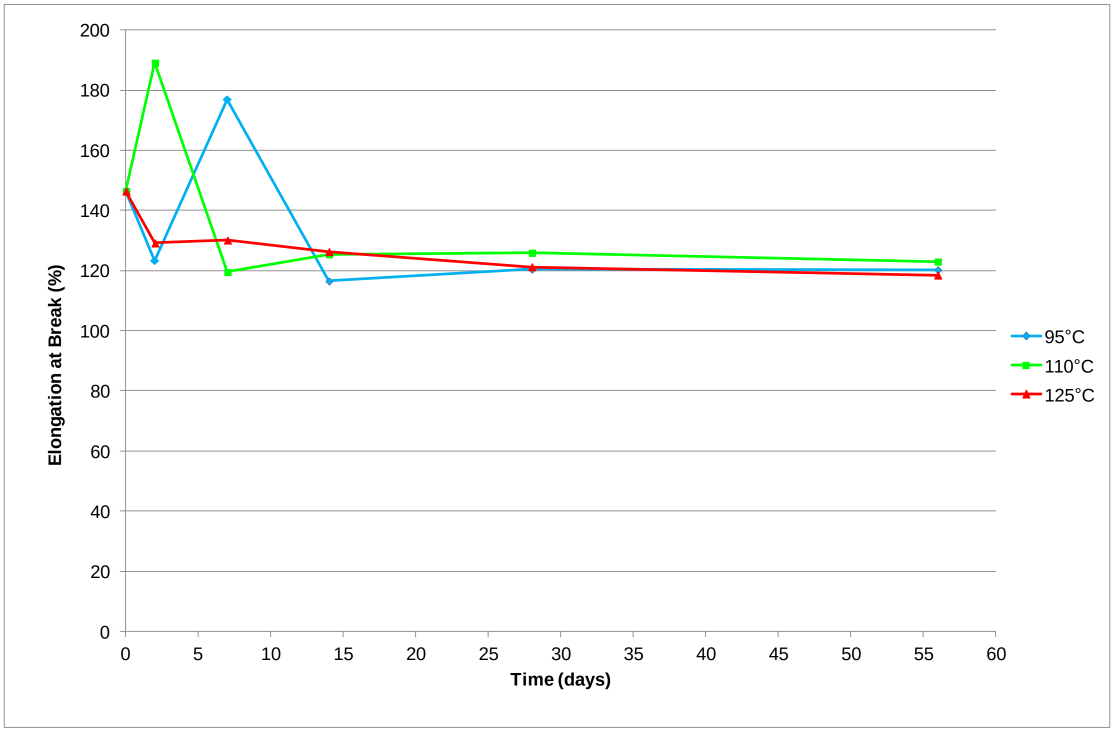

Figure C.7: N9036. Average elongation at break vs. fluid exposure time and temperature.