

Test Report no. 094/2007
18 April 2007
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Compatibility of Gylon with Perfluoropropane (C₃F₈)

Customer CERN EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH
CH-1211 GENEVE 23

Order No. CA 1476142

Responsible Dipl.-Ing. U. Grimm, Dipl.-Chem. M. Knabe
Tel (0351) 4081-750, Fax (0351) 4081-755

Material and methods

The compatibility of GYLON with octafluoropropane has to be tested in sealed container test. The material was provided by the customer.

Shouldered test bars of the material were stamped out. All were stamped out in the same direction. Weight after 30 min tempering at 60°C, dimensions, and Shore D hardness of the specimen were determined before storage in a 1 l sealed container. The container was closed, evacuated, and filled with 500 g octafluoropropane.

The sealed containers were tempered for 500 h at 60°C, which results in approximately 20 bar pressure.

After tempering the octafluoropropane was analysed by gas chromatography. The volatile contaminations were determined by GC-MS. The analyses were carried out using a HP 6890 system with mass sensitive detector MSD 5973.

Zertifiziert nach ISO 9001

The samples were injected with a gas loop. The following temperature programme was used: 5 min 50°C, 20 K/min until 180°C, 1 min 180°C. For the separation a 30 m capillary column J&W US3273715H GS-GASPRO was used.

After removing octafluoropropane the specimen were weighed, tempered for 30 min at 60°C, weighed again and measured.

To determine the extract the sealed container was cleaned with Methylene chloride, the solvent was given in a weighed glass beaker and the solvent evaporated. After that the beaker was weighed again.

The residue was investigated by IR-spectroscopie. For the investigation was used a FTIR- Spectrometer 4200 from Jasco.

Results

1. Optical valuation

The specimen were after the test more transparent like before the test (see fig 1).

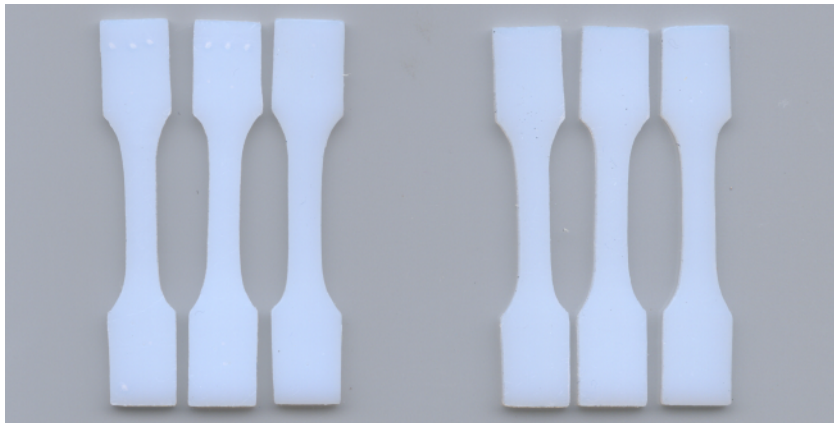


Fig. 1: specimen after test (left) and before test (right)

Sediments were found in the container.

2. Analyses

The gas chromatogram of octafluoropropane is given in fig. 2. Details can be found in fig. 3. Besides a main peak at retention time of 1.71 respectively 1.83 min there are several very small peaks in the sample.

The peaks are not integrated and not identified. Masses of CHF_2^- und CF_3^- were detected in the mass spectrum of the peaks.

The total amount of impurities is less than 0.01 %.

```
File       : C:\HPCHEM\1\DATA\KMAL185.D
Operator   : kn
Acquired   : 29 Mar 07 18:38      using AcqMethod QUALKM
Instrument  : GC/MS Ins
Sample Name: Cern 29.03.07
Misc Info  :
Vial Number: 1
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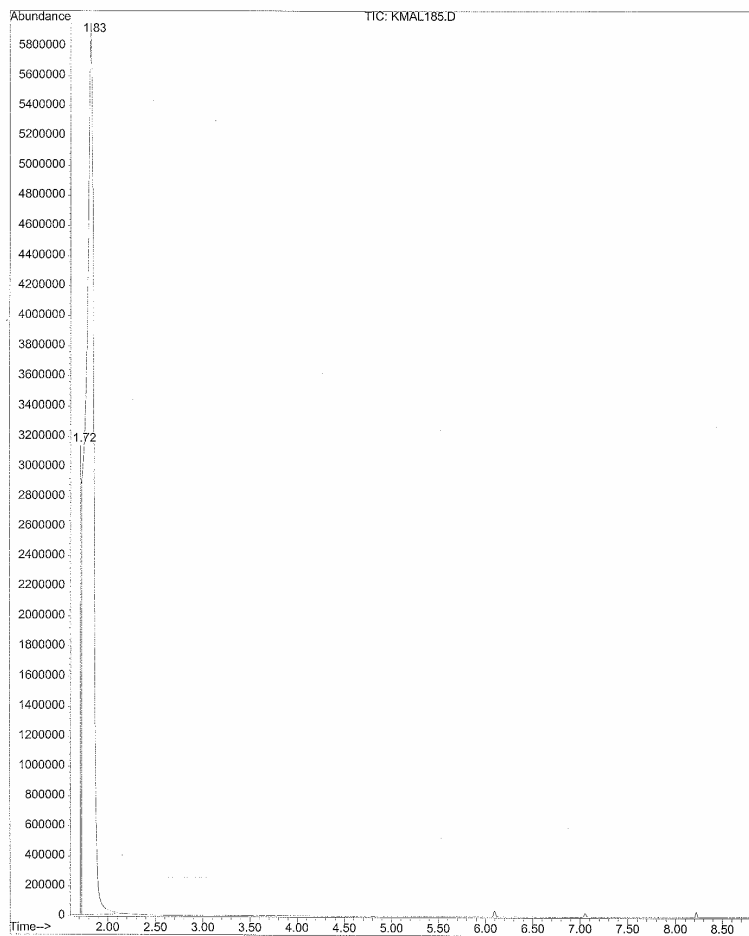


Figure 2

File : C:\HPCHEM\1\DATA\KMAL185.D
Operator : kn
Acquired : 29 Mar 07 18:38 using AcqMethod QUALKM
Instrument : GC/MS Ins
Sample Name : Cern 29.03.07
Misc Info :
Vial Number: 1

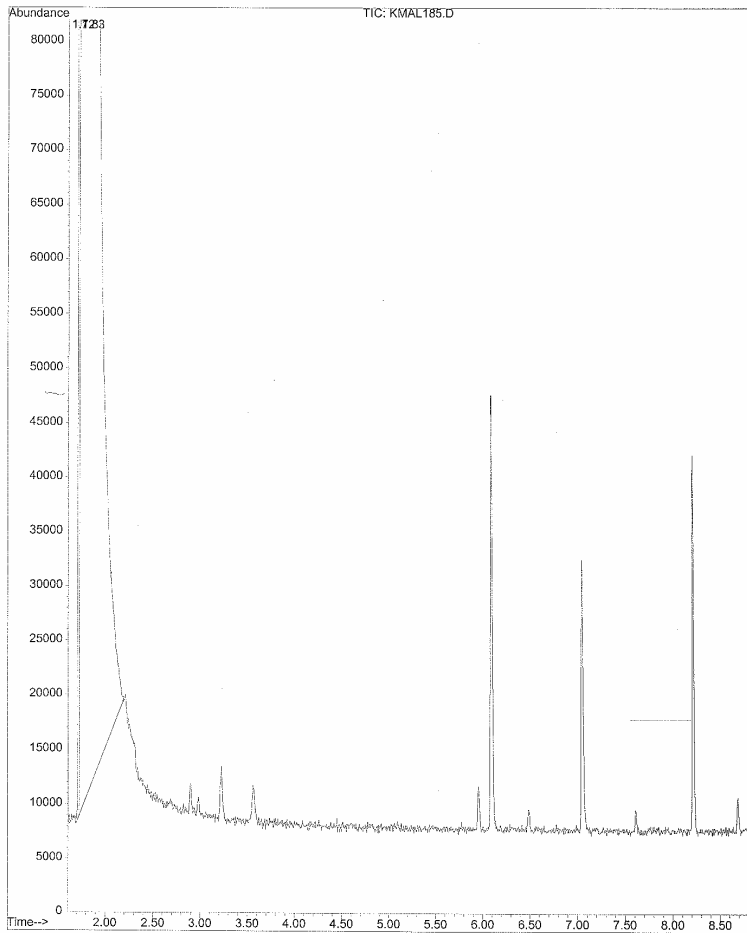


Figure 3

3. Changes in weight and dimensions

The results of weighting are given in table 1. The weight increase of the test bars after sealed container test was approximately 4.26 respectively 4.38 %.

Changes in dimension are given in table 2, 3, and 4. After sealed container test both length were increased about 1.12 % width of the specimen were increased about 1.97 %. There was an increase in thickness of the test bars of approximately 1.61 %.

The amount of the found extract was 6.2 mg and the mass of all specimen before the test was 13,747 g this gives a content of 0,05 %.

The IR-Spectrum of the extract is shown in figure 4. It is a mixture of organic compounds.

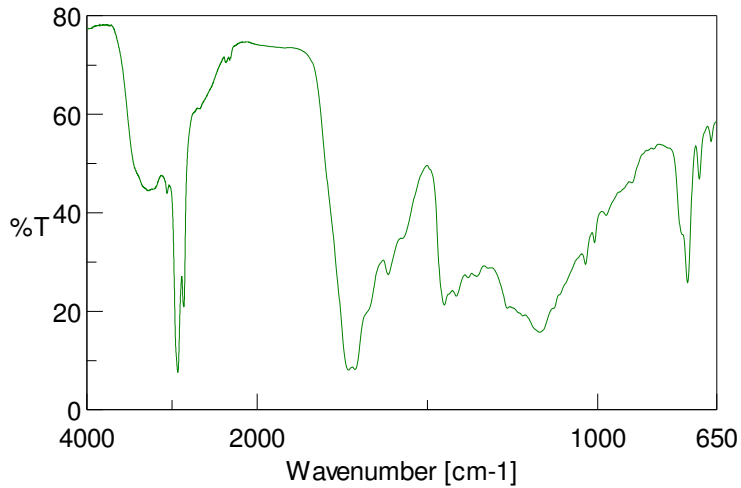


Figure 4

Table 1: Weights of specimen before and after sealed container tests

Sample No.	before sealed container test	immediately after sealed container test	Weight difference	after 30 min tempering at 60 °C	Weight difference
	[g]	[g]	[%]	[g]	[%]
1	1,3975	1,4588	4,39	1,4573	4,28
2	1,3843	1,4441	4,32	1,4430	4,25
3	1,3827	1,4428	4,34	1,4417	4,27
4	1,3606	1,4200	4,37	1,4183	4,24
5	1,3493	1,4084	4,38	1,4066	4,25
6	1,3797	1,4402	4,39	1,4386	4,27
7	1,3591	1,4185	4,37	1,4169	4,25
8	1,3847	1,4454	4,38	1,4436	4,25
9	1,3745	1,4355	4,44	1,4338	4,31
10	1,3746	1,4350	4,39	1,4332	4,26

Table 2: Length of specimen before and after sealed container tests

Sample No.	before sealed container test	after sealed container test	length difference
	[mm]	[mm]	[%]
1	50,34	51,02	1,35
2	50,37	50,89	1,03
3	50,36	50,91	1,09
4	50,47	51,21	1,47
5	50,57	51,33	1,50
6	50,88	51,21	0,65
7	50,85	51,30	0,88
8	50,58	51,12	1,07
9	50,63	51,18	1,09
10	50,88	51,41	1,04

Table 3: Width of specimen before and after sealed container tests

Sample No.	before sealed container test	after sealed container test	length difference
	[mm]	[mm]	[%]
1	4,22	4,32	2,37
2	4,21	4,29	1,90
3	4,20	4,28	1,90
4	4,18	4,22	1,0
5	4,12	4,17	1,21
6	4,16	4,27	2,64
7	4,14	4,18	1,0
8	4,16	4,28	2,88
9	4,14	4,26	2,90
10	4,21	4,29	1,90

Table 4: Thickness of specimen before and after sealed container tests

Sample No.	before sealed container test	after sealed container test	length difference
	[mm]	[mm]	[%]
1	1,91	1,95	2,09
2	1,91	1,95	2,09
3	1,92	1,94	1,04
4	1,89	1,92	0,69
5	1,89	1,92	0,69
6	1,91	1,94	1,57
7	1,88	1,93	2,66
8	1,91	1,94	1,57
9	1,91	1,94	1,57
10	1,90	1,94	2,11

4. Shore hardness

The results of Shore D hardness (DIN 53505) are listed in table 5. The Shore D hardness decreased about 18.47 %.

Table 5: Shore hardness of specimen before and after sealed container tests

sample No.	before sealed container test	after sealed container test	Difference [%]
1	51	42	17,65
2	53	43	18,87
3	52	43	17,31
4	53	44	16,98
5	53	43	18,87
6	52	43	17,31
7	53	43	18,87
8	53	42	20,75
9	52	42	19,23
10	53	43	18,87

5. Tensile Test

The tensile tests were carried out on a Universal testing machine SHIMADZU "Autograph" (updated by Zwick/Roell in Nov. 2006) with shouldered test bars (type S3A) according to the ISO 37:2005 at room temperature.

Load cell 1 kN, calibrated in April 2007.

No contacting optical extensometer (resolution: 10 μm , calibrated in March 2007)

The results for tensile strength and elongation are given in the appendix. The untreated specimens have an average tensile strength of 33.80 MPa with 3.00 MPa standard deviation. The elongation at fracture was 322 % with 19 % standard deviation. The specimens from the autoclave tests have an average tensile strength of 27.14 MPa with 1.19 MPa standard deviation and an elongation of 311 % with 11 % standard deviation.

Conclusions

After removing the specimen from the sealed container they showed a increase in weight, length, width and thickness and a large decrease in hardness.

An small amount of extract was founded.

There is a decrease of 24.5 % in tensile strength and a decrease in elongation at fracture of the specimen after sealed container test.

The material shows nearly no elastic behaviour in the refrigerant treated stage.

The stress-strain curve is changed after the test.

Octafluorpropane has an influence on the mechanical properties of Gylon.

Gylon is middling stable against octafluorpropane.












Dipl.-Chem. M. Knabe
Responsible co-worker

Dipl.-Ing. U. Grimm
Responsible co-worker

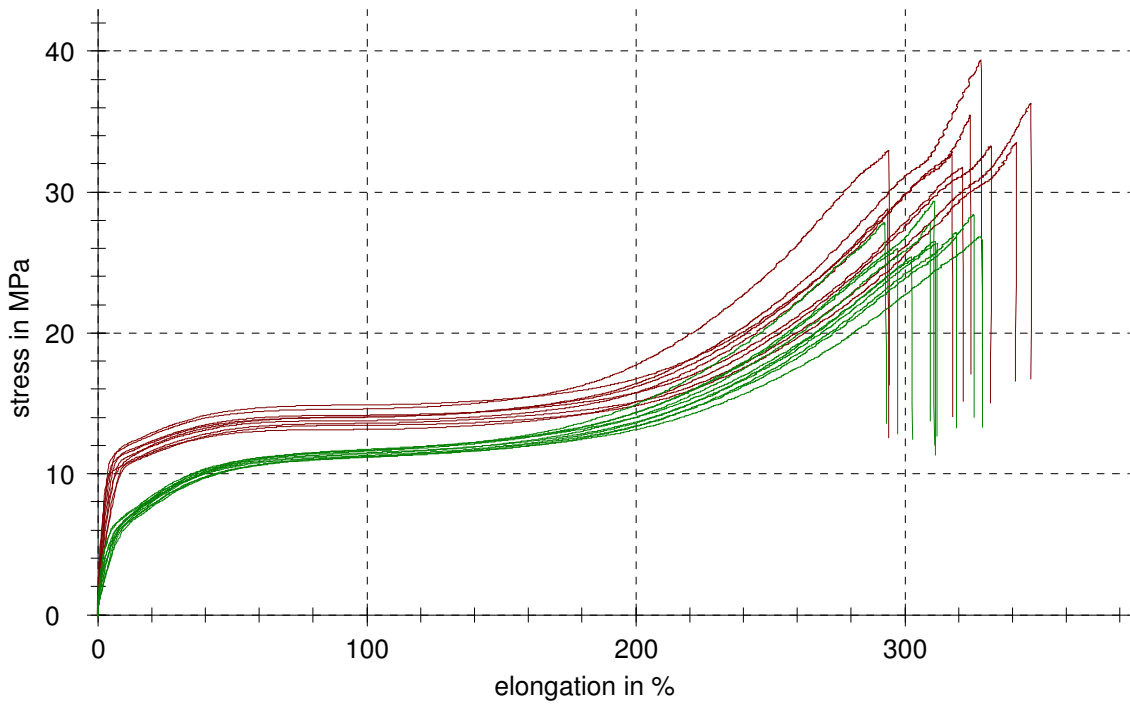
Testing environment:

Kunde : Client: CERN
 Prüfer : Tester: U. Grimm
 Prüfnorm : Testing standard: ISO 37:2005
 Versuchsablauf : Limit: 1000 N
 Testing speed: 200 mm/min; 0 s
 Material : Gylon
 Kraftaufnehmer : 1 kN
 Wegaufnehmer : optical
 Probenhalter : manuell
 Maschinendaten:

Single results:

Legende	Nr	Thickness a0 mm	Width b0 mm	L0 mm	$\sigma_{50\%}$ MPa	Bezeichn	$\sigma_{100\%}$ MPa	$\sigma_{200\%}$ MPa	$\sigma_{300\%}$ MPa	σ max MPa	ϵ max %
	1	2	4	10,02	12,86	new	13,16	15,09	27,58	33,27	332
	2	2	4	9,66	13,80		14,17	15,75	27,85	31,77	322
	3	2	4	9,98	13,58		14,06	16,20	-	28,79	294
	4	2	4	9,72	13,44		13,79	15,74	29,86	35,47	324
	5	2	4	10,06	13,67		14,01	16,39	31,09	39,38	328
	6	2	4	9,51	13,01		13,46	14,83	25,55	33,54	341
	7	2	4	10,10	14,24		14,60	17,69	-	32,96	294
	8	2	4	9,55	13,18		13,59	15,11	26,12	36,33	347
	9	2	4	9,72	14,48		14,90	16,75	29,80	32,64	317
	10	2	4	9,53	10,69	aged	11,74	13,98	25,03	25,40	302
	11	2	4	9,63	10,62		11,49	13,67	24,69	26,37	312
	12	2	4	9,91	10,83		11,67	14,35	-	26,00	297
	13	2	4	9,63	10,55		11,41	14,02	25,81	27,73	309
	14	2	4	9,59	10,32		11,20	13,41	23,85	28,36	326
	15	2	4	9,92	10,80		11,68	14,90	-	27,83	292
	16	2	4	9,62	10,27		11,17	13,36	24,21	27,11	319
	17	2	4	10,00	10,73		11,61	14,36	26,76	29,36	311
	18	2	4	9,29	10,28		11,23	13,13	22,70	26,81	328
	19	2	4	9,69	10,47		11,30	13,61	24,91	26,47	311

Results graphs:



Statistics:

new n = 9	Thickness a0 mm	Width b0 mm	L0 mm	$\sigma_{50\%}$ MPa	$\sigma_{100\%}$ MPa	$\sigma_{200\%}$ MPa	$\sigma_{300\%}$ MPa	σ_{max} MPa	ϵ_{max} %
x	2	4	9,81	13,58	13,97	15,95	28,26	33,80	322
s	0,000	0,000	0,23	0,54	0,55	0,92	2,06	3,00	19
v	0,00	0,00	2,32	3,97	3,93	5,74	7,30	8,87	5,75

aged n = 10	Thickness a0 mm	Width b0 mm	L0 mm	$\sigma_{50\%}$ MPa	$\sigma_{100\%}$ MPa	$\sigma_{200\%}$ MPa	$\sigma_{300\%}$ MPa	σ_{max} MPa	ϵ_{max} %
x	2	4	9,68	10,56	11,45	13,88	24,75	27,14	311
s	0,000	0,000	0,21	0,21	0,22	0,55	1,23	1,19	11
v	0,00	0,00	2,19	2,01	1,90	3,95	4,97	4,38	3,69