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Compatibility of Kel-F (PCTFE) with C_3F_8

Customer	CERN
Order No.	DAI 1717027
Responsible	Dr.-Ing. M. Junk, Dipl.-Chem. M. Knabe Tel (0351) 4081-750, Fax (0351) 4081-755

Material and methods

The compatibility of PCTFE with octafluorpropane has to be tested in sealed container test.
Shouldered test bars of the material were provided by the customer.
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 145 g octafluorpropane.

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

After tempering the octafluorpropane 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.

Institut für Luft- und Kältetechnik gemeinnützige Gesellschaft mbH • Bertolt-Brecht-Allee 20 • 01309 Dresden
Geschäftsführer: Dr. rer. nat. habil. Ralf Herzog • Prokurist: Prof. Dr.-Ing. Uwe Franzke
Tel.: 0351/4081-520 • Fax 0351/4081-525 • E-Mail: gf@ilkdresden.de • http://www.ilkdresden.de
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The samples were injected with a gas loop. 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 octafluorpropane the specimen were weighed, tempered for 30 min at 60°C, weighed again and measured.

Results

1. Optical valuation

Visible changes of the specimen were not detected.
Sediments were not found in the container.

2. Analyses

The gas chromatogram of octafluorpropane is given in fig. 1. Details can be found in fig. 2 and 3. Besides a main peak at retention time of 2.97 respectively 3.29 min there are four smaller peaks in the sample.
They are related to:

1.	air	RT 1.3 min
2.	CO_2	RT 1.8 min
3.	octafluorpentane	RT 7.8 min
4.	not identified	RT 7.9 min (Since masses of CHF_2- und CF_3- were detected in the mass spectrum the substance is a fluorocarbon.)

The total amount of impurities is less than 0.01 %. Fluorocarbons are caused by production.
The peak at 9.753 min is methylene chloride and comes not from the sample. Methylene chloride was used to clean the valve between the GC and the sealed container.

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 0.027%.

Changes in dimension are given in table 2, 3, and 4. After sealed container test both length and width of the specimen were shrunken about 0.33%. There was an increase in thickness of the test bars of approximately 0,77%.

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

Sample No.	before sealed container test	immediately after sealed container test	after 30 min tempering at 60°C	Weight difference
	[g]	[g]	[g]	[%]
1	19.0183	19.0308	19.0274	0.048
2	19.0730	19.0842	19.0744	0.007
3	19.0522	19.0577	19.0694	0.090
4	19.3639	19.372	19.3654	0.008
5	18.4417	18.4532	18.4452	0.019
6	18.8135	18.8175	18.8154	0.010
7	18.7700	18.7730	18.7713	0.007

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	150.20	149.69	0.34
2	150.20	149.72	0.32
3	150.28	149.79	0.33
4	150.07	149.57	0.33
5	150.21	149.71	0.33
6	150.35	149.86	0.33
7	150.45	149.96	0.33

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	20.05	19.99	0.29
2	19.95	19.86	0.35
3	20.02	19.94	0.40
4	19.91	19.87	0.20
5	19.89	19.79	0.40
6	20.23	20.16	0.34
7	20.00	19.93	0.35

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	4.29	4.31	0.47
2	4.3	4.31	0.23
3	4.29	4.33	0.93
4	4.37	4.41	0.91
5	4.15	4.20	1.20
6	4.22	4.26	0.95
7	4.22	4.25	0.71

4. Shore hardness and tensile tests

The results of Shore D hardness (DIN 53505) are listed in table 5. Both, before and after testing in C_3F_8 the hardness of the samples is 78 Shore D.

The tensile tests were carried out on a Shimadzu test machine AG-100kNG according to EN ISO 527 Parts 1 and 2 with shouldered test bars (type 1B) at room temperature with 10 mm/min test speed.

The results for tensile strength σ_m and elongation ε_B are given in table 6. The untreated specimens have an average tensile strength of 49.9 MPa with 1.3 MPa standard deviation. The elongation at

fracture was 188.1% with 47.7% standard deviation. The specimens from the autoclave tests have an average tensile strength of 54.3 MPa with 0.5 MPa standard deviation and an elongation of 142.7% with 45.2% standard deviation. Both samples before (Fig. 4) and after (Fig. 5) sealed container test have a stress-strain-curve with a yield point.

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

sample No.	before sealed container test	after sealed container test
1	77	77
2	78	78
3	78	78
4	78	78
5	78	78
6	78	78
7	78	78

Table 6: Tensile strength and elongation of specimen before and after sealed container tests

sample No.	before sealed container test		after sealed container test	
	σ_m [MPa]	ε_B [%]	σ_m [MPa]	ε_B [%]
1	51.6	84.97	54.12	181.2
2	48.64	183.9	54.53	168.1
3	51.19	205.1	53.64	52.88
4	48.61	197.5	54.6	146.1
5	50.81	218.1	54.44	165.4
6	48.64	198.8	55.1	170
7	50.03	228	54.0	115

Conclusions

After removing the specimen from the sealed container they showed only a small increase in weight and thickness and a small decrease in length and width. There is a slight increase in tensile strength and a noticeable decrease in elongation at fracture of the specimen after sealed container test.

On test conditions given above PCTFE shows a good resistance against octafluorpropane.

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

Dr.-Ing. M. Junk
Responsible co-worker

Area Percent Report

Data File : C:\HPCHEM\1\DATA\KMT46.D
Acq On : 27 Sep 04 1:08 pm
Sample : Oktofluorpropan n. Vers.
Misc :

Vial: 1
Operator: kn
Inst : GC/MS Ins
Multiplr: 1.00
Sample Amount: 0.00

MS Integration Params: events.e
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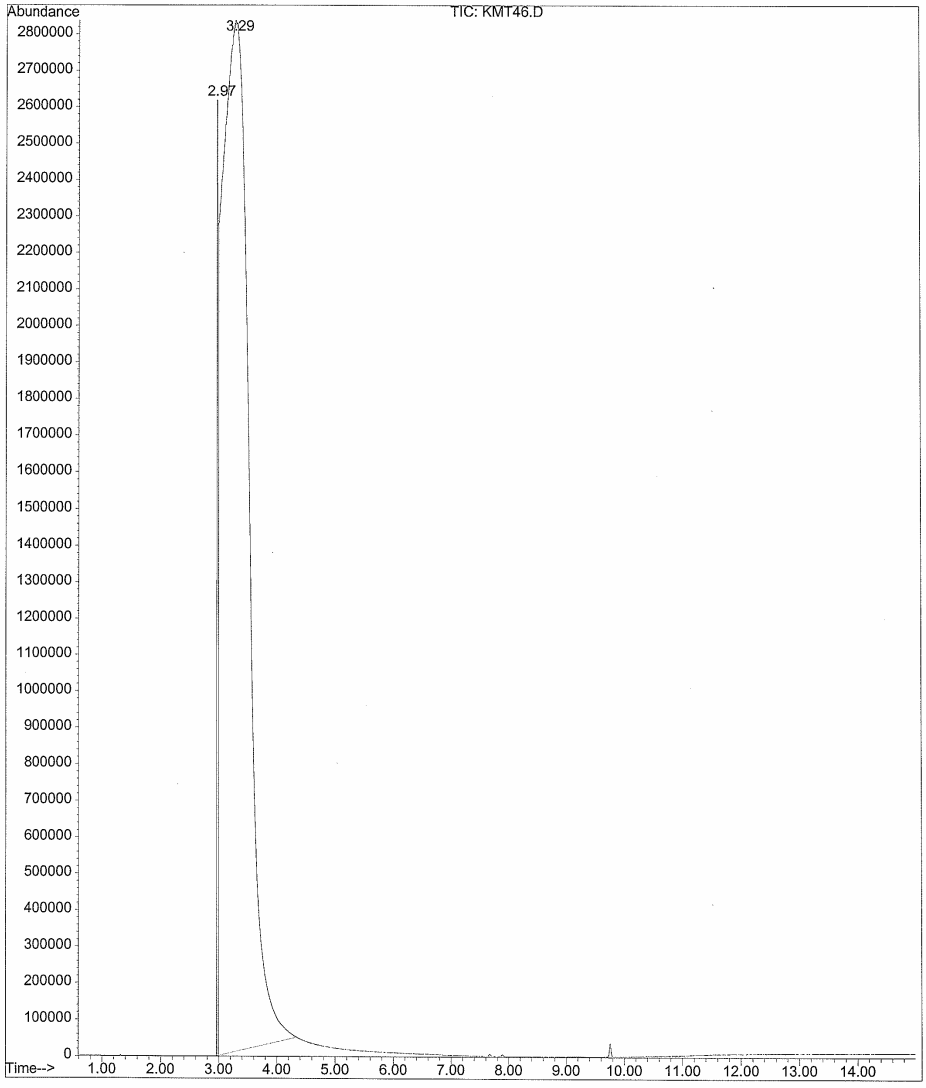
Title : detar



KMT46.D HFKWN2.M Mon Sep 27 13:29:55 2004

Fig. 1: Gas chromatogram of octafluorpropane

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Sample Name: Oktofluorpropan n. Vers.
Misc Info   :
Vial Number: 1
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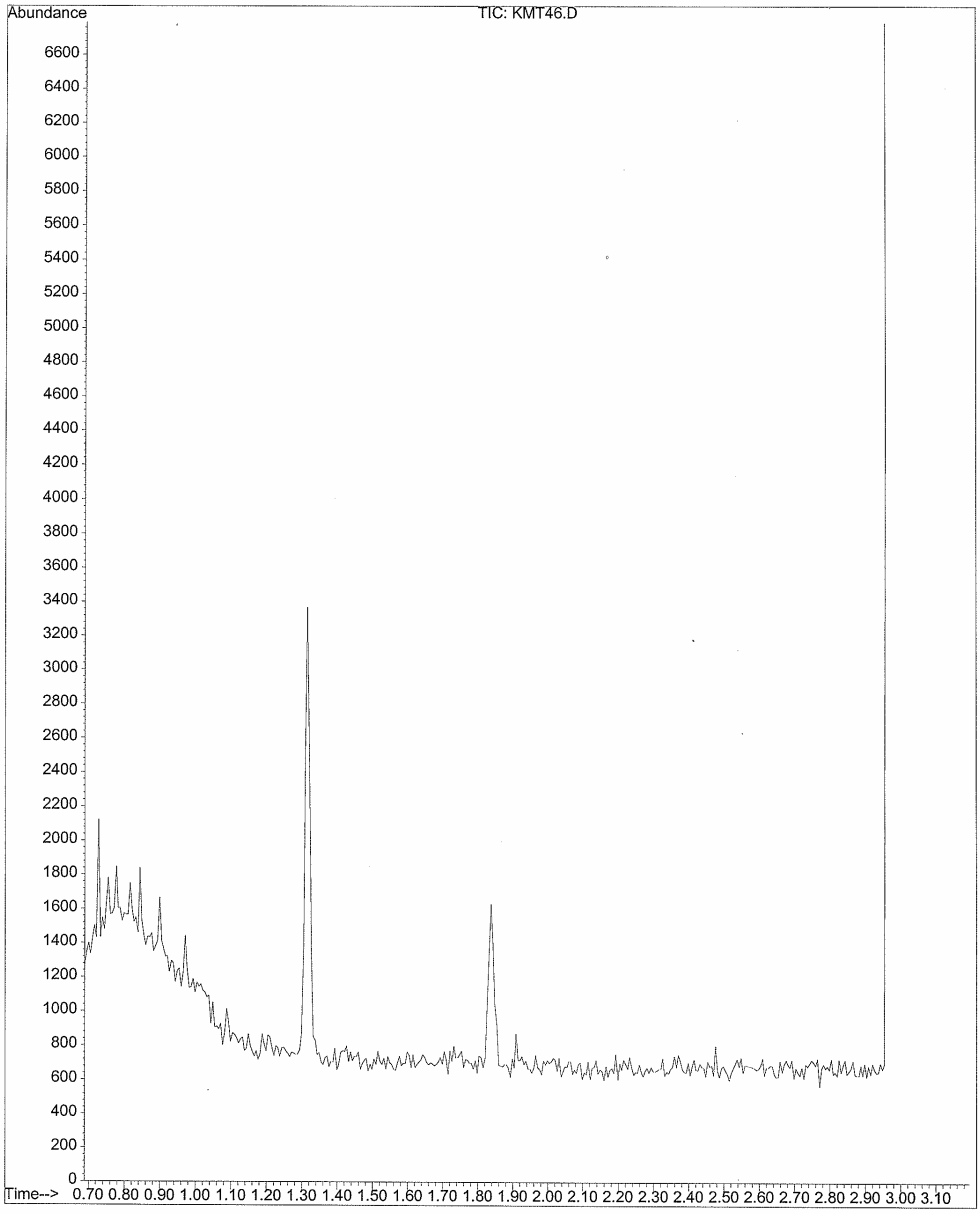

Fig. 2: detail from fig. 1

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Operator    : kn
Acquired    : 27 Sep 04  13:08     using AcqMethod HFKWN2
Instrument  :   GC/MS Ins
Sample Name: Oktofluorpropan n. Vers.
Misc Info   :
Vial Number: 1
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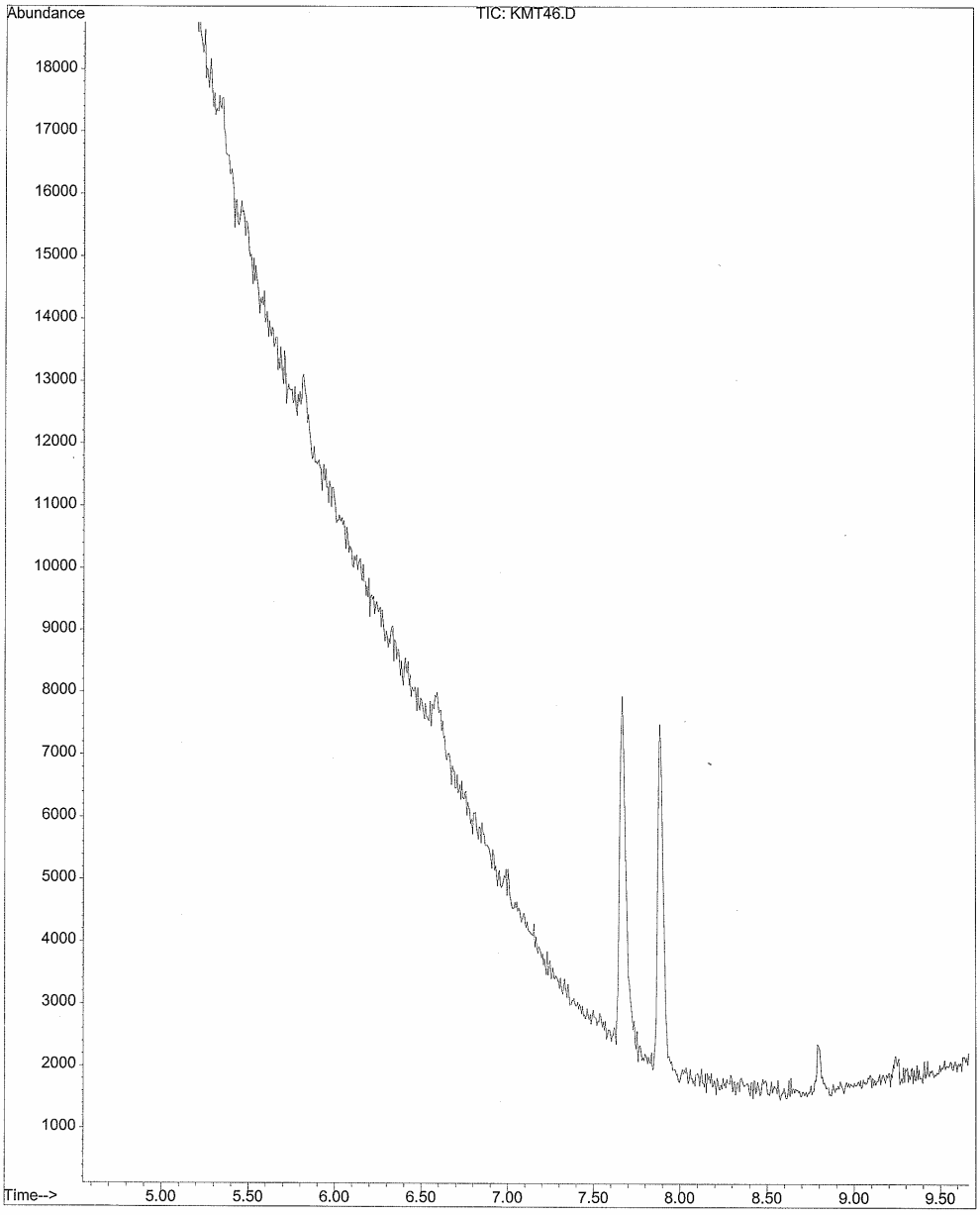

Fig. 3: detail from fig. 1

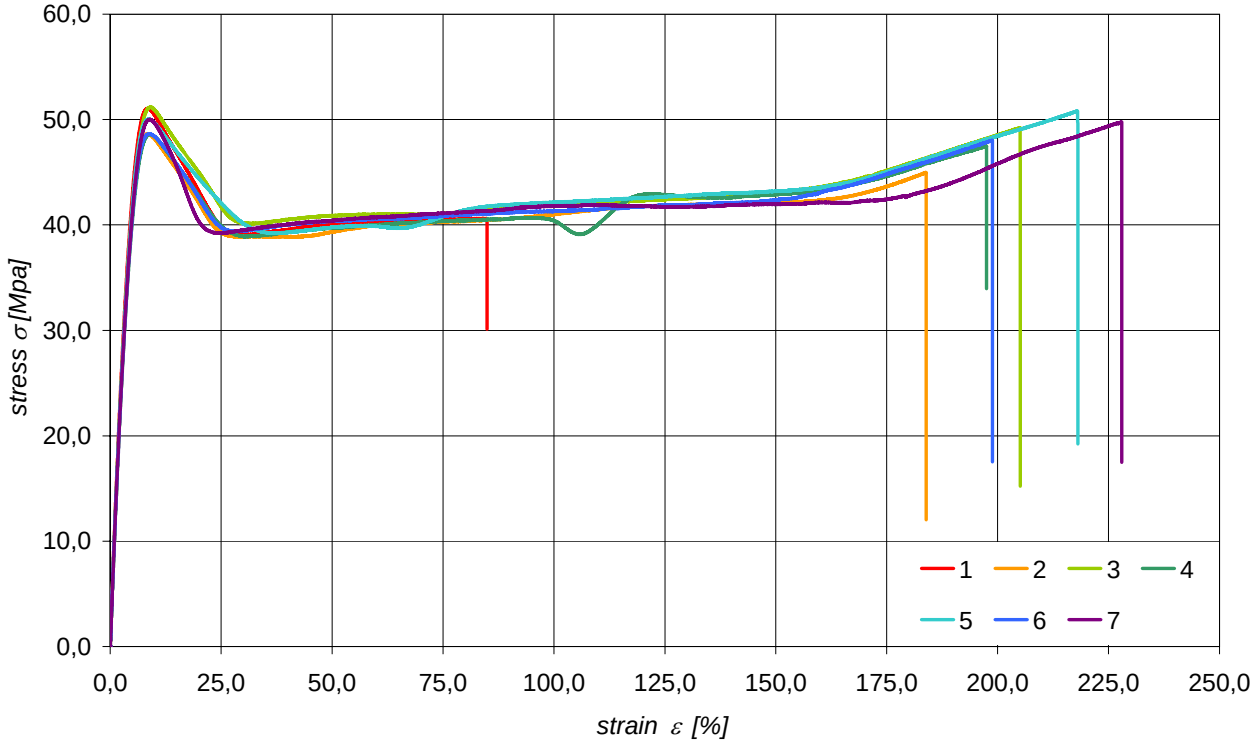

Fig. 4: stress-strain-diagram of Kel-F (PCTFE)

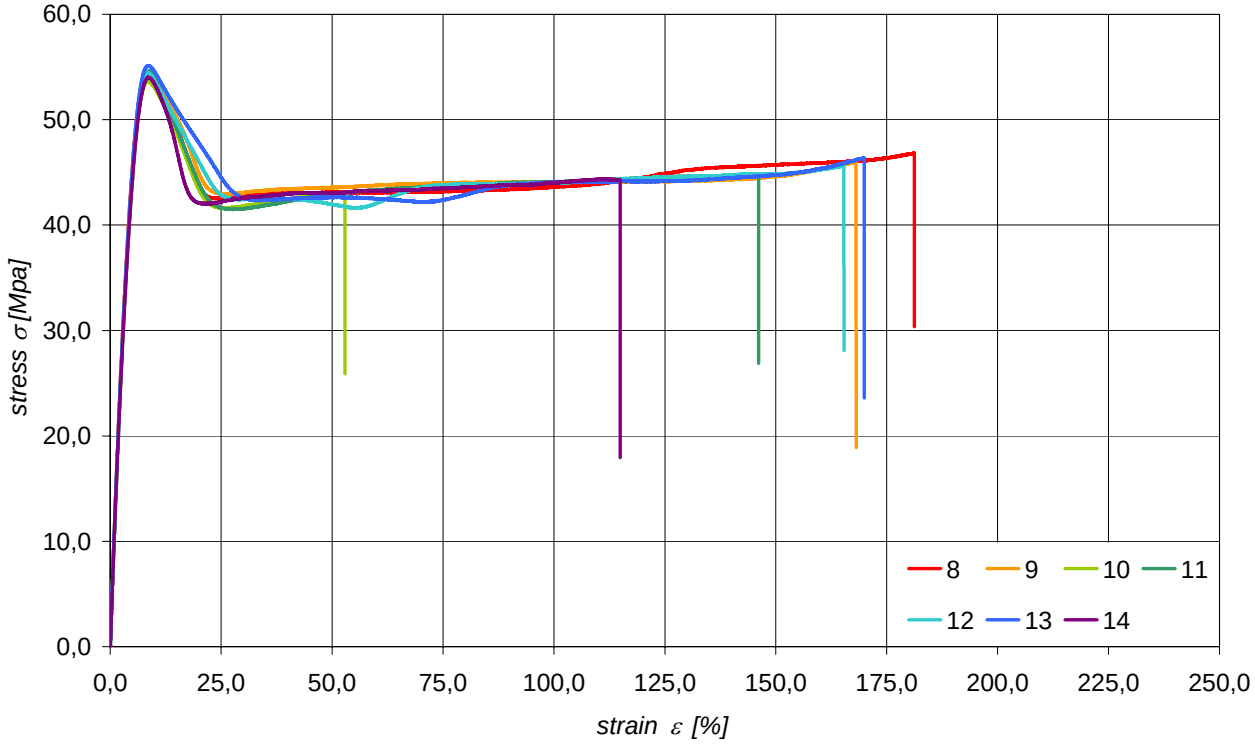

Fig. 5: stress-strain-diagram of Kel-F (PCTFE) after sealed container tests