SIGRAFLEX® Foil
Approvals/Test reports

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effective May 2018, superseding all previous versions

Additional information on our SIGRAFLEX® sealing materials can be found in the Download Center on our homepage.

DIN-DVGW-Baumusterprüfzertifikat

DIN-DVGW type examination certificate

NG-5124AQ1478
Registriernummer
registration number

Anwendungsbereich ***field of application***	Produkte der Gasversorgung *products of gas supply*
Zertifikatinhaber ***owner of certificate***	SGL CARBON GmbH Werner-von-Siemens-Straße 18, D-86405 Meitingen
Vertreiber ***distributor***	SGL CARBON GmbH Werner-von-Siemens-Straße 18, D-86405 Meitingen
Produktart ***product category***	Schmier-/Dicht-/Betriebsmittel: Flachdichtungswerkstoff auf Basis Graphit (5124)
Produktbezeichnung ***product description***	Flachdichtungswerkstoff auf Basis Graphit
Modell ***model***	®SIGRAFLEX unverstärkt
Prüfberichte ***test reports***	Baumusterprüfung: 14/342/5124/1 vom 27.02.2015 (EBI)
Prüfgrundlagen ***test basis***	DIN 3535-6 (01.01.2011)

Ablaufdatum / AZ ***date of expiry / file no.***	28.02.2020 / 14-0784-GNV

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DVGW CERT GmbH is an accredited body by DAkkS according to EN 45011:1998 for certification of products for energy and water supply industry.

DAkkS
Deutsche Akkreditierungsstelle
D-ZE-16028-01-01

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TEST REPORT

On Testing a Nonmetallic Material for Reactivity with Gaseous and Liquid Oxygen

BAM reference	15019178 I E
Copy	1st copy of 2 copies
Customer	SGL Carbon GmbH Werner-von-Siemens-Straße 20 86405 Meitingen Germany
Order date	March 23, 2015
Reference	Order No.: 112-10-45827456
Receipt of order	April 1, 2015
Test samples	Sigraflex® Foil Z, undisclosed batch; BAM Order-No.: 2.1/52 578
Receipt of samples	April 2, 2015
Test date	April 16 to December 4, 2015
Test location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120
Test procedure according to	ISO 21010:2014 and DIN EN 1797:2002-02 "Cryogenic Vessels - Gas/Material Compatibility" Annex of code of practice M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry, Edition: March 2014; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen", Edition: June 2013
Safety Related Maximum Operating Conditions	See chapter 4 "Summary and Evaluation"

All pressures of this report are excess pressures.
This test report consists of page 1 to 8 and annexes 1 to 5.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
"Testing and evaluating the compatibility of the nonmetallic material Sigraflex® Foil Z, undisclosed batch, for use as a sealing material in components and flanged connections for gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar as well as for use in liquid oxygen service"

1 Safety Data Sheet
(7 pages, version 2.0.0, date of issue: June 4, 2014)

20 Disks of graphite based Sigraflex® Foil Z, undisclosed batch
Outer-Ø: 140 mm; Thickness: 0.5 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of the nonmetallic material Sigraflex® Foil Z, undisclosed batch, for use as a sealing material in gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar, testing of ignition sensitivity to gaseous oxygen impacts at 60 °C, 250 °C and 300 °C, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance, and a flange test were carried out.

The compatibility of the material with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1.

Results:

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	150	no reaction*
60	1	170	no reaction*
60	1	300	no reaction*
60	1	350	no reaction*
60	1	450	ignition on 5. impact
60	1	430	no reaction*
60	1	440	ignition on 4. impact
60	1	430	no reaction*
250	1	430	no reaction*
250	1	430	no reaction*
300	1	430	no reaction*
300	1	430	ignition on 3. impact
300	1	410	no reaction*
300	1	420	no reaction*
300	1	420	ignition on 5. impact
300	1	410	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reaction of the test sample with oxygen could be detected at a final oxygen pressure p_F of 430 bar and at temperatures of 60 °C and 250 °C.

In two separate tests, each consisting of a series of five consecutive impacts, no reaction of the test sample with oxygen could be detected at a final oxygen pressure p_F of 410 bar and at a temperature of 300 °C.

3.2 Autogenous Ignition Temperature (AIT)

Based on the specified maximum operating pressure of the nonmetallic material, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 160 bar. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	62	162	> 500
2	62	164	> 500
3	62	164	> 500
4	62	166	> 500
5	62	163	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of p_I = 62 bar. The final oxygen pressure p_F was approximately 164 bar. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.3 Artificial Aging

In general, the aging test is carried out at the maximum operating pressure and at an elevated temperature, which is 25 °C above the maximum operating temperature. In this case, the aging test was carried out at 160 bar and at 325 °C. The test method is described in annex 3.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	325	160	- 0.2

After aging of the test sample at 325 °C and at 160 bar oxygen pressure, the test sample was apparently unchanged. The sample lost 0.2 % in mass.

3.3.1 AIT after Artificial Aging

The same test conditions as in chapter 3.2 were used for determining the autogenous ignition temperature after aging. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	62	165	> 500
2	62	164	> 500
3	62	164	> 500
4	62	165	> 500
5	62	163	> 500

Up to temperatures of 500 °C, no ignition of the aged sample could be detected in five tests with initial oxygen pressures of p_I = 62 bar. The final oxygen pressure p_F was approximately 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.4 Flange Test

Based on the specified maximum operating conditions, flange testing was performed at 160 bar oxygen pressure and at a temperature of 300 °C. The test method is described in annex 4.

Results:

Test Number	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	160	300	Only those parts of the gasket burn that project into the pipe, the flange connection remains gas-tight
2	160	300	same behavior as in test no. 1
3	160	300	same behavior as in test no. 1
4	160	300	same behavior as in test no. 1
5	160	300	same behavior as in test no. 1

In five tests at 160 bar oxygen pressure and 300 °C, only those parts of the gasket burn that project into the pipe; the fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange remains gas-tight.

3.5 Reactivity with Liquid Oxygen on Mechanical Impact

In general, a nonmetallic material is not compatible with liquid oxygen, if reactions occur at a drop height of 0.17 m (impact energy 125 Nm) or less. The test method is described in annex 5.

Results:

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	0.83	625	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	no reaction
5	1.00	750	no reaction
6	1.00	750	no reaction
7	1.00	750	no reaction
8	1.00	750	no reaction
9	1.00	750	no reaction
10	1.00	750	no reaction
11	1.00	750	no reaction

At a drop height of 1.00 m (impact energy 750 Nm), in ten separate tests, no reaction of the nonmetallic material with liquid oxygen could be detected.

4 Summary and Evaluation

No autogenous ignition temperature could be determined up to 500 °C as no ignition of Sigraflex® Foil Z, undisclosed batch, could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 164 bar. The AIT in high pressure oxygen can only be determined up to 500 °C. That equals the maximum operating temperature of the test equipment.

At a temperature of 325 °C and an oxygen pressure of 160 bar, the material proved to be aging resistant. The sample was apparently unchanged and lost 0.2 % in mass.

No AIT of the aged material, would be determined up to 500 °C, as no ignition could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

For safety reasons a safety margin of 100 °C between AIT and maximum operating temperature is being considered in evaluating nonmetallic materials for oxygen service. As the maximum operating temperature of the nonmetallic material is 300 °C, Sigraflex® Foil Z, undisclosed batch, fulfills this criterion.

According to DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010: 2014 „Cryogenic Vessels - Gas/Material Compatibility" the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use Sigraflex® Foil Z, undisclosed batch, as a sealing material in gaseous oxygen service at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	430
> 60 up to 300	160

In addition and based on the results of the flange test, there are no objections with regard to technical safety, to use Sigraflex® Foil Z, undisclosed batch, as a gasket material with a maximum thickness of 0.5 mm in gaseous oxygen service in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
300	160

This applies to flat faces flanges, male/female flanges, and flanges with tongue and groove.

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", described in annex 5, there are also no objections with regard to technical safety to use Sigraflex® Foil Z, undisclosed batch, in liquid oxygen service. In this case, a limitation to a particular pressure range is not necessary as compression of liquid oxygen causes no significant change in concentration and therefore has no considerable influence on the reactivity of the material.

5 Comments

This evaluation is based exclusively on the results of the tested batch of the nonmetallic material Sigraflex® Foil Z.

Products on the market that contain a reference to BAM testing shall be marked accordingly. It shall be evident that only a sample of a batch has been tested and evaluated for oxygen compatibility. The reference shall not produce a presumption of conformity that monitoring of the production on a regular basis is being performed by BAM.

It shall be clear that the product may only be used for gaseous and liquid oxygen service. The maximum safe oxygen pressure of the product and its maximum use temperature as well as other restrictions in use shall be given.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

January 7, 2016

Division 2.1
"Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list:
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TEST REPORT

On Testing a Nonmetallic Material for Reactivity with Gaseous and Liquid Oxygen

BAM reference	15019178 III E
Copy	1st copy of 2 copies
Customer	SGL Carbon GmbH Werner-von-Siemens-Straße 20 86405 Meitingen Germany
Order date	March 23, 2015
Reference	Order No.:112-10-45827456
Receipt of order	April 1, 2015
Test samples	Sigraflex® APX2, undisclosed batch; BAM Order-No.: 2.1/52 578
Receipt of samples	April 2, 2015
Test date	April 16 to December 4, 2015
Test location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120
Test procedure according to	ISO 21010:2014 and DIN EN 1797:2002-02 "Cryogenic Vessels - Gas/Material Compatibility" Annex of code of practice M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry, Edition: March 2014; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen", Edition: June 2013
Safety Related Maximum Operating Conditions	See chapter 4 "Summary and Evaluation"

All pressures of this report are excess pressures.
This test report consists of page 1 to 8 and annexes 1 to 5.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
"Testing and evaluating the compatibility of the nonmetallic material Sigraflex® APX2, undisclosed batch, for use as a sealing material in components and flanged connections for gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar as well as for use in liquid oxygen service"

1 Safety Data Sheet
(5 pages, version 1.0.1, date of issue: July 25, 2014)

20 Disks of graphite based Sigraflex® APX2, undisclosed batch
Outer-Ø: 140 mm; Thickness: 0.5 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of the nonmetallic material Sigraflex® APX2, undisclosed batch, for use as a sealing material in gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar, testing of ignition sensitivity to gaseous oxygen impacts at 60 °C, 250 °C and 300 °C, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance, and a flange test were carried out.

The compatibility of the material with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1.

Results:

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	300	no reaction*
60	1	400	no reaction*
60	1	450	no reaction*
60	1	450	no reaction*
250	1	450	no reaction*
250	1	450	no reaction*
300	1	450	no reaction*
300	1	450	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reaction of the test sample with oxygen could be detected at a final oxygen pressure p_F of 450 bar and at temperatures of 60 °C, 250 °C and 300 °C. Testing to ignition sensitivity to gaseous oxygen impacts can only be performed up to 450 bar, that equals the maximum working pressure of the test equipment.

3.2 Autogenous Ignition Temperature (AIT)

Based on the specified maximum operating pressure of the nonmetallic material, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 160 bar. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	62	164	> 500
2	62	165	> 500
3	62	164	> 500
4	62	164	> 500
5	62	165	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of p_I = 62 bar. The final oxygen pressure p_F was approximately 164 bar. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.3 Artificial Aging

In general, the aging test is carried out at the maximum operating pressure and at an elevated temperature, which is 25 °C above the maximum operating temperature. In this case, the aging test was carried out at 160 bar and at 325 °C. The test method is described in annex 3.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	325	160	- 1.4

After aging of the test sample at 325 °C and at 160 bar oxygen pressure, the test sample was apparently unchanged. The sample lost 1.4 % in mass.

3.3.1 AIT after Artificial Aging

The same test conditions as in chapter 3.2 were used for determining the autogenous ignition temperature after aging. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	62	165	> 500
2	62	165	> 500
3	62	165	> 500
4	62	164	> 500
5	62	164	> 500

Up to temperatures of 500 °C, no ignition of the aged sample could be detected in five tests with initial oxygen pressures of p_I = 62 bar. The final oxygen pressure p_F was approximately 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.4 Flange Test

Based on the specified maximum operating conditions, flange testing was performed at 160 bar oxygen pressure and at a temperature of 300 °C. The test method is described in annex 4.

Results:

Test Number	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	160	300	Only those parts of the gasket burn that project into the pipe, the flange connection remains gas-tight
2	160	300	same behavior as in test no. 1
3	160	300	same behavior as in test no. 1
4	160	300	same behavior as in test no. 1
5	160	300	same behavior as in test no. 1

In five tests at 160 bar oxygen pressure and 300 °C, only those parts of the gasket burn that project into the pipe; the fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange remains gas-tight.

3.5 Reactivity with Liquid Oxygen on Mechanical Impact

In general, a nonmetallic material is not compatible with liquid oxygen, if reactions occur at a drop height of 0.17 m (impact energy 125 Nm) or less. The test method is described in annex 5.

Results:

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	1.00	750	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	no reaction
5	1.00	750	no reaction
6	1.00	750	no reaction
7	1.00	750	no reaction
8	1.00	750	no reaction
9	1.00	750	no reaction
10	1.00	750	no reaction

At a drop height of 1.00 m (impact energy 750 Nm), in ten separate tests, no reaction of the nonmetallic material with liquid oxygen could be detected.

4 Summary and Evaluation

No autogenous ignition temperature (AIT) could be determined up to 500 °C as no ignition of Sigraflex® APX2, undisclosed batch, could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 164 bar. The AIT in high pressure oxygen can only be determined up to 500 °C. That equals the maximum operating temperature of the test equipment.

At a temperature of 325 °C and an oxygen pressure of 160 bar, the material proved to be sufficient aging resistant. The sample was apparently unchanged and lost 1.4 % in mass.

No AIT of the aged material would be determined up to 500 °C, as no ignition could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

For safety reasons a safety margin of 100 °C between AIT and maximum operating temperature is being considered in evaluating nonmetallic materials for oxygen service. As the maximum operating temperature of the nonmetallic material is 300 °C, Sigraflex® APX2, undisclosed batch, fulfills this criterion.

According to DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010: 2014 „Cryogenic Vessels - Gas/Material Compatibility" the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C. Testing to ignition sensitivity to gaseous oxygen impacts can only be performed up to 450 bar, that equals the maximum working pressure of the test equipment.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use Sigraflex® APX2, undisclosed batch, as a sealing material in gaseous oxygen service at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	450
> 60 up to 300	160

In addition and based on the results of the flange test, there are no objections with regard to technical safety, to use Sigraflex® APX2, undisclosed batch, as a gasket material with a maximum thickness of 0.5 mm in gaseous oxygen service in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
300	160

This applies to flat faces flanges, male/female flanges, and flanges with tongue and groove.

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", described in annex 5, there are also no objections with regard to technical safety to use Sigraflex® APX2, undisclosed batch, in liquid oxygen service. In this case, a limitation to a particular pressure range is not necessary as compression of liquid oxygen causes no significant change in concentration and therefore has no considerable influence on the reactivity of the material.

5 Comments

This evaluation is based exclusively on the results of the tested batch of the nonmetallic material Sigraflex® APX2.

Products on the market that contain a reference to BAM testing shall be marked accordingly. It shall be evident that only a sample of a batch has been tested and evaluated for oxygen compatibility. The reference shall not produce a presumption of conformity that monitoring of the production on a regular basis is being performed by BAM.

It shall be clear that the product may only be used for gaseous and liquid oxygen service. The maximum safe oxygen pressure of the product and its maximum use temperature as well as other restrictions in use shall be given.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

January 7, 2016

Division 2.1
"Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list: 1st copy: SGL Carbon GmbH
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TEST REPORT

On Testing a Nonmetallic Material for Reactivity with Gaseous and Liquid Oxygen

BAM reference	15019178 II E
Copy	1st copy of 2 copies
Customer	SGL Carbon GmbH Werner-von-Siemens-Straße 20 86405 Meitingen Germany
Order date	March 23, 2015
Reference	Order No.: 112-10-45827456
Receipt of order	April 1, 2015
Test samples	Sigraflex® APX, undisclosed batch; BAM Order-No.: 2.1/52 578
Receipt of samples	April 2, 2015
Test date	April 16 to December 4, 2015
Test location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120
Test procedure according to	ISO 21010:2014 and DIN EN 1797:2002-02 "Cryogenic Vessels - Gas/Material Compatibility" Annex of code of practice M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry, Edition: March 2014; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen", Edition: June 2013
Safety Related Maximum Operating Conditions	See chapter 4 "Summary and Evaluation"

All pressures of this report are excess pressures.
This test report consists of page 1 to 8 and annexes 1 to 5.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
"Testing and evaluating the compatibility of the nonmetallic material Sigraflex® APX, undisclosed batch, for use as a sealing material in components and flanged connections for gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar as well as for use in liquid oxygen service"

1 Safety Data Sheet
(5 pages, version 1.0.1, date of issue: July 25, 2014)

20 Disks of graphite based Sigraflex® APX, undisclosed batch
Outer-Ø: 140 mm; Thickness: 0.5 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of the nonmetallic material Sigraflex® APX, undisclosed batch, for use as a sealing material gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar, testing of ignition sensitivity to gaseous oxygen impacts at 60 °C, 250 °C and 300 °C, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance, and a flange test were carried out.

The compatibility of the material with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1.

Results:

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	150	no reaction*
60	1	180	no reaction*
60	1	250	no reaction*
60	1	300	ignition on 1. impact
60	1	290	ignition on 4. impact
60	1	280	no reaction*
60	1	280	no reaction*
250	1	280	no reaction*
250	1	280	no reaction*
300	1	280	no reaction*
300	1	280	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reaction of the test sample with oxygen could be detected at a final oxygen pressure p_F of 280 bar and at temperatures of 60 °C, 250 °C and 300 °C.

3.2 Autogenous Ignition Temperature (AIT)

Based on the specified maximum operating pressure of the nonmetallic material, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 160 bar. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	62	165	> 500
2	62	165	> 500
3	62	165	> 500
4	62	165	> 500
5	62	164	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of p_I = 62 bar. The final oxygen pressure p_F was approximately 165 bar. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.3 Artificial Aging

In general, the aging test is carried out at the maximum operating pressure and at an elevated temperature, which is 25 °C above the maximum operating temperature. In this case, the aging test was carried out at 160 bar and at 325 °C. The test method is described in annex 3.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	325	160	- 1.2

After aging of the test sample at 325 °C and at 160 bar oxygen pressure, the test sample was apparently unchanged. The sample lost 1.2 % in mass.

3.3.1 AIT after Artificial Aging

The same test conditions as in chapter 3.2 were used for determining the autogenous ignition temperature after aging. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	62	166	> 500
2	62	164	> 500
3	62	166	> 500
4	62	165	> 500
5	62	161	> 500

Up to temperatures of 500 °C, no ignition of the aged sample could be detected in five tests with initial oxygen pressures of p_I = 62 bar. The final oxygen pressure p_F was approximately 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.4 Flange Test

Based on the specified maximum operating conditions, flange testing was performed at 160 bar oxygen pressure and at a temperature of 300 °C. The test method is described in annex 4.

Results:

Test Number	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	160	300	Only those parts of the gasket burn that project into the pipe, the flange connection remains gas-tight
2	160	300	same behavior as in test no. 1
3	160	300	same behavior as in test no. 1
4	160	300	same behavior as in test no. 1
5	160	300	same behavior as in test no. 1

In five tests at 160 bar oxygen pressure and 300 °C, only those parts of the gasket burn that project into the pipe; the fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange remains gas-tight.

3.5 Reactivity with Liquid Oxygen on Mechanical Impact

In general, a nonmetallic material is not compatible with liquid oxygen, if reactions occur at a drop height of 0.17 m (impact energy 125 Nm) or less. The test method is described in annex 5.

Results:

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	1.00	750	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	severe
5	0.83	625	no reaction
6	0.83	625	no reaction
7	0.83	625	no reaction
8	0.83	625	severe
9	0.67	500	no reaction
10	0.67	500	no reaction
11	0.67	500	no reaction
12	0.67	500	no reaction
13	0.67	500	no reaction
14	0.67	500	no reaction
15	0.67	500	no reaction
16	0.67	500	no reaction
17	0.67	500	no reaction
18	0.67	500	no reaction

At a drop height of 0.67 m (impact energy 500 Nm), in ten separate tests, no reaction of the nonmetallic material with liquid oxygen could be detected.

4 Summary and Evaluation

No autogenous ignition temperature (AIT) could be determined up to 500 °C as no ignition of Sigraflex® APX, undisclosed batch, could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 165 bar. The AIT in high pressure oxygen can only be determined up to 500 °C. That equals the maximum operating temperature of the test equipment.

At a temperature of 325 °C and an oxygen pressure of 160 bar, the material proved to be sufficient aging resistant. The sample was apparently unchanged and lost 1.2 % in mass.

No AIT of the aged material, would be determined up to 500 °C, as no ignition could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

For safety reasons a safety margin of 100 °C between AIT and maximum operating temperature is being considered in evaluating nonmetallic materials for oxygen service. As the maximum operating temperature of the nonmetallic material is 300 °C, Sigraflex® APX, undisclosed batch, fulfills this criterion.

According to DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010: 2014 „Cryogenic Vessels - Gas/Material Compatibility" the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use Sigraflex® APX, undisclosed batch, as a sealing material in gaseous oxygen service at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	280
> 60 up to 300	160

In addition and based on the results of the flange test, there are no objections with regard to technical safety, to use Sigraflex® APX, undisclosed batch, as a gasket material with a maximum thickness of 0.5 mm in gaseous oxygen service in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
300	160

This applies to flat faces flanges, male/female flanges, and flanges with tongue and groove.

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", described in annex 5, there are also no objections with regard to technical safety to use Sigraflex® APX, undisclosed batch, in liquid oxygen service. In this case, a limitation to a particular pressure range is not necessary as compression of liquid oxygen causes no significant change in concentration and therefore has no considerable influence on the reactivity of the material.

5 Comments

This evaluation is based exclusively on the results of the tested batch of the nonmetallic material Sigraflex® APX.

Products on the market that contain a reference to BAM testing shall be marked accordingly. It shall be evident that only a sample of a batch has been tested and evaluated for oxygen compatibility. The reference shall not produce a presumption of conformity that monitoring of the production on a regular basis is being performed by BAM.

It shall be clear that the product may only be used for gaseous and liquid oxygen service. The maximum safe oxygen pressure of the product and its maximum use temperature as well as other restrictions in use shall be given.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

January 7, 2016

Division 2.1
"Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list: 1st copy: SGL Carbon GmbH
2nd copy: BAM - Division 2.1 "Gases, Gas Plants"

Report

on Testing a Nonmetallic Material for Reactivity with Oxygen

Bundesanstalt für Materialforschung und -prüfung

Reference Number	2-1432/2014 E
Copy	1. Copy of 2 Copies
Customer	SGL Carbon GmbH Werner-von-Siemens-Str. 18 86405 Meitingen Germany
Order Date	June 10, 2014, and order extension of July 15, 2014
Reference	Delivery No.: 31015513
Receipt of Order	June 12 and July 15, 2014
Test Samples	Sigraflex® Folie Typ E, undisclosed batch, for use as a sealing material in piping, valves and fittings or other components at temperatures up to 250 °C and for use as a gasket material for gaseous oxygen service at temperatures up to 250 °C and 130 bar and for liquid oxygen service; BAM Order-No.: 2.1/52 150
Receipt of Samples	June 11, 2014
Test Date	July 2 to September 5, 2014
Test Location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120
Test Procedure or Requirement According to	DIN EN 1797: 2002-02 "Cryogenic Vessels - Gas/Material Compatibility" ISO 21010: 2004-07 "Cryogenic Vessels - Gas/Material Compatibility" Annex of pamphlet M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen by BAM Federal Institute for Material Research and Testing.", by Berufsgenossenschaft Rohstoffe und chemische Industrie, Edition: September 2011 TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen" Edition: June 2013

All pressures of this report are excess pressures.
This test report consists of page 1 to 7 and annex 1 to 5.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test Application
1 Material Data Sheet (2 pages, date of issue: 03/2011)
1 Safety Data Sheet
(7 pages, revision no. 1.02, date of issue: April 24, 2014)
15 Disks Sigraflex® Folie Typ E, undisclosed batch
Outer-Ø: 140 mm; Thickness: 0.5 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of Sigraflex® Folie Typ E, undisclosed batch, for use as a sealing material in piping, valves and fittings or other components for gaseous oxygen service at temperatures up to 250 °C, the ignition sensitivity to gaseous oxygen impacts at 60 °C, at 200°C and at 250 °C, a determination of the autogenous ignition temperature (AIT) and an investigation of the aging resistance in high pressure oxygen were carried out.

To evaluate the compatibility of Sigraflex® Folie Typ E, undisclosed batch, for use as a gasket material for gaseous oxygen service at temperatures up to 250 °C and 130 bar, in addition a flange test at 250 °C and 130 bar was carried out.

The compatibility of Sigraflex® Folie Typ E, undisclosed batch, with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1.

Results:

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	450	no reaction*
60	1	450	ignition on 1. impact
60	1	440	ignition on 4. impact
60	1	430	ignition on 2. impact
60	1	420	no reaction*
60	1	420	ignition on 1. impact

* within a series of five consecutive impacts

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	410	no reaction*
60	1	410	ignition on 1. impact
60	1	400	no reaction*
60	1	400	ignition on 1. impact
60	1	390	no reaction*
60	1	390	ignition on 1. impact
60	1	380	ignition on 3. impact
60	1	370	no reaction*
60	1	370	ignition on 1. impact
60	1	360	no reaction*
60	1	360	ignition on 1. impact
60	1	350	ignition on 1. impact
60	1	340	ignition on 2. impact
60	1	320	no reaction*
60	1	320	ignition on 1. impact
60	1	300	no reaction*
60	1	300	ignition on 1. impact
60	1	280	no reaction*
60	1	280	ignition on 1. impact
60	1	270	no reaction*
60	1	270	no reaction*
200	1	270	no reaction*
200	1	270	no reaction*
250	1	270	no reaction*
250	1	270	ignition on 1. impact
250	1	250	no reaction*
250	1	260	ignition on 4. impact
250	1	250	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of Sigraflex® Folie Typ E, undisclosed batch, with oxygen could be observed at a final oxygen pressure p_F of 270 bar and at temperatures of 60 °C and 200 °C.

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of Sigraflex® Folie Typ E, undisclosed batch, with oxygen could be observed at a final oxygen pressure p_F of 250 bar and at a temperature of 250 °C.

3.2 Autogenous Ignition Temperature (AIT)

As the maximum operating pressure of Sigraflex® Folie Typ E, undisclosed batch, for use as a gasket material is 130 bar and in accordance with the costumer, the test was performed at a final oxygen pressure of approximately 130 bar. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	50	134	> 500
2	50	133	> 500
3	50	134	> 500
4	50	134	> 500
5	50	133	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of p_I = 50 bar. The final oxygen pressure p_F was approximately 133 bar.

3.3 Artificial Aging

The test method is described in annex 3.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	275	130	± 0

After aging of the test sample at 275 °C and 130 bar oxygen pressure, the test sample was apparently unchanged. The mass of the test sample did not change.

3.3.1 AIT after Artificial Aging

The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	50	131	> 500
2	50	133	> 500
3	50	133	> 500
4	50	134	> 500
5	50	131	> 500

Up to temperatures of 500 °C, no ignition of the aged sample could be detected in five tests with initial oxygen pressures of p_I = 50 bar. The final oxygen pressure p_F was approximately 132 bar. This shows, that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

3.4 Flange Test

According to the above-mentioned operating conditions of Sigraflex® Folie Typ E, undisclosed batch, for use as a gasket material the flange test was performed at 130 bar oxygen pressure and 250 °C. The test method is described in annex 4.

Results:

Test No.	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	130	250	Only those parts of the gasket burn that project into the pipe. The flange remains gas-tight.
2	130	250	Same behavior as in test no. 1
3	130	250	Same behavior as in test no. 1
4	130	250	Same behavior as in test no. 1
5	130	250	Same behavior as in test no. 1

In five tests at 130 bar oxygen pressure and 250 °C, only those parts of the gasket burn that project into the pipe; the fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange remains gas-tight.

3.5 Reactivity with Liquid Oxygen on Mechanical Impact

In general, a nonmetallic material is not compatible with liquid oxygen, if reactions occur at a drop height of 0.17 m (impact energy 125 Nm) or less. The test method is described in annex 5.

Results:

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	0.83	625	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	no reaction
5	1.00	750	no reaction
6	1.00	750	no reaction
7	1.00	750	no reaction
8	1.00	750	no reaction
9	1.00	750	no reaction
10	1.00	750	no reaction
11	1.00	750	no reaction

At drop heights of 1.00 m (impact energy 750 Nm), in ten separate tests, no reaction of the test sample with liquid oxygen could be detected

4 Summary and Evaluation

Up to temperatures of 500 °C, no ignition of the nonmetallic material Sigraflex® Folie Typ E, undisclosed batch, could be detected in five tests with final oxygen pressures of approximately p_F = 133 bar.

At a temperature of 275 °C and an oxygen pressure of 130 bar, the material proved to be aging resistant. The mass of the test sample did not change.

Up to temperatures of 500 °C, no ignition of the aged material could be detected in five tests with final oxygen pressures of approximately p_F = 132 bar. This shows, that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

Generally, in evaluating nonmetallic materials for oxygen service, a safety margin of 100 °C between AIT and maximum operating temperature is being considered for safety reasons. As the maximum operating temperature is 250 °C, Sigraflex® Folie Typ E, undisclosed batch, fulfills this criterion.

According to DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010: 2004-07 „Cryogenic Vessels - Gas/Material Compatibility" the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use Sigraflex® Folie Typ E, undisclosed batch, as a sealing material in piping, valves and fittings, or other components for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	270
> 60 to 250	130

On basis of the test results and the results of the flange test, there are also no abjections with regard to technical safety, to use Sigraflex® Folie Typ E, undisclosed batch, as a gasket material with a maximum thickness of 0.5 mm in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	130

This applies to flat faced flanges, male/female flanges, and flanges with tongue and groove.

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", described in annex 5, there are also no objections with regard to technical safety to use Sigraflex® Folie Typ E, undisclosed batch, in valves and fittings or other components for liquid oxygen service. In this case, a limitation to a particular pressure range is not necessary as compression of liquid oxygen causes no significant change in concentration and therefore has no considerable influence on the reactivity of the material.

5 Comments

The test results refer exclusively to the tested batch of Sigraflex® Folie Typ E, undisclosed batch.

Products on the market that contain a reference to BAM testing shall be marked accordingly. It shall be evident that only a sample of a batch has been tested and evaluated for oxygen compatibility. The reference shall not produce a presumption of conformity that monitoring of the production on a regular basis is being performed by BAM.

For safety reason, it is not justifiable to use our BAM reference number without additional information about the purpose of use and the maximum operating conditions.

It shall be clear that the product may only be used for gaseous oxygen service and liquid oxygen service. The maximum safe oxygen pressure of the product and its maximum operating temperature as well as other restrictions in use shall be given.

BAM Federal Institute for Materials Research and Testing
12200 Berlin, September 15, 2014

Division 2.1
"Gases, Gas Plants"

On behalf of

Dr. Thomas Kasch

Copies: 1. Copy: SGL Carbon GmbH
2. Copy: BAM - Division 2.1 "Gases, Gas Plants"

TZW
Prüfstelle Wasser

PRÜFZEUGNIS (V)

über die Untersuchung von "SIGRAFLEX® FOLIE" gemäß KTW-Leitlinie des Umweltbundesamtes (UBA)

Hersteller:	SGL CARBON GmbH, Meitingen
Art der Proben:	Graphitqualität
Bezeichnung der Proben:	"SIGRAFLEX® FOLIE"
Art der Prüfkörper:	Probeplatten
Eingang der Proben:	13.05.2008
Probenehmer:	Auftraggeber
TZW-Az.:	KA 0010/13

Untersuchungsergebnisse

1. Rezeptur: wurde unter KC 103/13 vorgelegt und überprüft
2. Migrationstest:

Kaltwasser 23°C	1. – 3. Tag	4. – 6. Tag	7. – 9. Tag	Richtwert für 3. Extraktion
Klarheit, Färbung, Geruch, Geschmack, Schaumbildung	nnb	nnb	nnb	nicht nennenswert beeinflusst
C-Abgabe [mg C/m²d]	< 1	< 1	< 1	≤ 125
Cl_2-Zehrung [mg Cl_2/m²d]	19	10	7	

DC

Die untersuchten Proben "SIGRAFLEX® FOLIE" entsprechen den Anforderungen der KTW-Leitlinie des Umweltbundesamtes (Bgesundhbl. 2005) im Bereich Dichtungen.

Anmerkung:

Dieses Prüfzeugnis basiert auf der Erstprüfung (TZW-Az.: KA 182A/08) vom 24.06.2008.
Die Gültigkeit dieses Prüfzeugnisses richtet sich nach andernorts festgelegten Bestimmungen. Sie endet jedoch spätestens am 23.06.2018.

Karlsruhe, den 20.02.2013

R. Turki

Dr. J. Klinger / i.A. Dr.-Ing. R. Turkovic
Leiter der Prüfstelle

Das Technologiezentrum Wasser ist eine Einrichtung des DVGW *Deutscher Verein des Gas- und Wasserfaches e.V.* – Technisch-wissenschaftl. Verein –

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pruefstelle@tzw.de, www.tzw.de

Herstellerbescheinigung
Manufacturer's Certificate

Die SGL CARBON GmbH als Lieferant der Graphitfolienmaterialien

SGL CARBON GmbH as supplier of the graphite foil materials

SIGRAFLEX® Folie C
SIGRAFLEX® Folie E
SIGRAFLEX® Folie APX
SIGRAFLEX® Folie Z

bestätigt, dass diese Materialien der Verordnung (EG) Nr. 1935/2004 über Materialien und Gegenstände, die dazu bestimmt sind, mit Lebensmitteln in Berührung zu kommen, entspricht.
confirms, that these materials comply with the Regulation (EC) No. 1935/2004 on materials and articles intended to come into contact with food.

Meitingen, 24. Febr. 2014

SGL CARBON GmbH
Arbeitssicherheit und Umweltschutz
Environment, Health and Safety

Dr. Meyer zu Reckendorf

Approval Number: 1603506
Test Report: MA5572/J

Water Regulations Advisory Scheme Ltd.
Unit 13,
Willow Road,
Pen y Fan Industrial Estate,
Crumlin,
Gwent,
NP11 4EG

16th March 2016

SGL Carbon GmbH
Laboratory Services,
Drachenburgstraße 1,
53170 Bonn,
Germany

WATER REGULATIONS ADVISORY SCHEME LTD. (WRAS)
MATERIAL APPROVAL

The material referred to in this letter is suitable for contact with wholesome water for domestic purposes having met the requirements of BS6920-1:2000 and/or 2014 'Suitability of non-metallic products for use in contact with water intended for human consumption with regard to their effect on the quality of the water'.

The reference relates solely to its effect on the quality of the water with which it may come into contact and does not signify the approval of its mechanical or physical properties for any use.

GRAPHITE - MATERIAL ONLY. **5135**

`SIGRAFLEX® APX`. Grey coloured, expansion/compression moulded graphite material. For use with water up to 85°C.

APPROVAL NUMBER: 1603506
APPROVAL HOLDER: SGL CARBON GMBH

The Scheme reserves the right to review approval.
Approval 1603506 is valid between March 2016 and March 2021

An entry, as above, will accordingly be included in the Water Fittings Directory on-line under the section headed, "Materials which have passed full tests of effect on water quality".

The Directory may be found at: www.wras.co.uk/directory

Yours faithfully

Jason Furnival
Approvals & Enquiries Manager
Water Regulations Advisory Scheme

WRAS MATERIAL APPROVAL - MATERIALS WHICH HAVE PASSED FULL TESTS OF EFFECT ON WATER QUALITY

The material referred to in this letter is suitable for contact with water for domestic purposes. **Approval of this material does not signify the approval of its mechanical or physical properties for any use.**

Manufacturers or applicants may only quote in their sales literature terms which are used in this letter, namely that; 'the material as listed, having passed the tests of effect on water quality, is suitable for use in contact with wholesome water'

This may be abbreviated to 'Water Regulations Advisory Scheme - Approved Material' or 'WRAS Approved Material'.

The scope of an Approval does not extend to rebranded materials unless otherwise agreed by the Scheme.

Use of the WRAS Approved Material Logo

Approval holders may use the WRAS Approved Material logo and make reference to any approval issued by WRAS Ltd. in respect of a particular material or range of materials provided the approval is, and remains valid.

Approval holders are entitled to use the logo on the packing, promotional literature and point of sale advertising Approved Materials.

Modifications to existing Approvals

It is a condition of WRAS Material Approval that NO changes or modifications to the Approved Material, be made without the Approval Holder first notifying WRAS Ltd. Full details of the proposed changes must be provided to the Scheme. Failure to comply with this condition will immediately invalidate a previously granted Approval.

Re-Approval

WRAS will write to you 1 year before the approval expires asking whether you would like to renew it. Please complete the relevant section of the MA3 application form which will be included with the letter and return to WRAS (via e-mail or post).

Please note it is the responsibility of the Approval Holder to ensure the Approval remains valid. WRAS Ltd. accepts no liability for the delay in granting approval where this is caused by circumstances outside of the Scheme's control.

LGA QualiTest GmbH
Chemische Produktprüfung / *Chemical Product Testing*

TÜVRheinland®
LGA

Zertifiziert nach DIN EN ISO 9001/14001

Prüfbericht / *Test Report*

L G A QualiTest GmbH • Postfach 3022 • 90014 Nürnberg

SGL Carbon GmbH
Postfach 1193
D-86400 Meitingen

5681826-AT1

Auftraggeber / *Orderer*:	wie Adressat / *see consignee*
Auftrag vom / *Order Date*:	15.09.2008
Prüfgegenstand / *Test Specimen*:	Graphit-Folie / *graphite foil* *SIGRAFLEX F05010Z*
Inhalt des Auftrages / *Scope*:	Chemische Prüfung / *Chemical Test*
Eingang der Proben / *Samples Received*:	15.09.2008
Untersuchungszeitraum / *Testing Period*:	17.09. bis / *until* 07.10.2008

Untersuchungsergebnis / *Test Result:*

Prüfanforderungen für / *test-requirements for:*	
Abgabe von Schwermetallen / *Release of Heavy Metals*	erfüllt / *passed*
Abgabe von Cyanid / *Release of Cyanide*	erfüllt / *passed*
Farblässigkeit / *Transfer of Colourants*	erfüllt / *passed*
Gesamtmigration / *Total Migration*	erfüllt / *passed*
Polycyclische aromatische Kohlenwasserstoffe (PAK) / *Polycyclic aromatic Hydrocarbons (PAH)*	erfüllt / *passed*
Phthalatweichmacher in Bedarfsgegenständen mit Lebensmittelkontakt / *Phthalate Softeners in Food Contact Materials*	erfüllt / *passed*
Sensorische Prüfung / *Sensory Test*	erfüllt / *passed*

(Details siehe nachfolgenden Bericht / *for details see the following report*)

LGA QualiTest GmbH • TÜV Rheinland Group
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Geschäftsführer:
Hans-Hermann Ueffing, Michael F. Jungnitsch
Steuer-Nr. 241/115/90768 Ust-IdNr. DE813835603

LGA QualiTest GmbH
Chemische Produktprüfung / *Chemical Product Testing*

TÜVRheinland®
LGA

Prüfbericht Nr. /
test report no. 5681826-AT1.doc

Probenbezeichnung / *Tested Materials*

Abgabe von Schwermetallen / *Release of Heavy Metals*

Untersuchungsverfahren / *Test Method*

Migrationsbedingungen / *migration conditions* :
3 % Essigsäure / *3 % acetic acid*, 10 d, 40°C
Bestimmung mittels ICP-MS. / *Determination by means of ICP-MS.*

Untersuchungsergebnisse / *Test Results*

Probe / *sample*	Cr	Ni	Pb	Cd	Bewertung / *Assessment*
Graphit-Folie / *graphite foil*	< 0,01 mg/dm²	< 0,01 mg/dm²	< 0,01 mg/dm²	< 0,001 mg/dm²	erfüllt / *passed*

Probe / *sample*	Cd	Sb	As	Hg	Bewertung / *Assessment*
Graphit-Folie / *graphite foil*	< 0,001 mg/dm²	< 0,001 mg/dm²	< 0,001 mg/dm²	< 0,001 mg/dm²	erfüllt / *passed*

Probe / *sample*	Se	Zn
Graphit-Folie / *graphite foil*	< 0,001 mg/dm²	< 0,001 mg/dm²

Eine Abgabe an toxischen Schwermetallen war nicht bzw. nur in gesundheitlich unbedenklichen Spuren feststellbar. Die Zusammensetzung von Lebensmitteln wird dadurch nicht beeinträchtigt. / *A release of toxic heavy metals was not detectable respectively only in harmless traces. The composition of foodstuffs will not be affected.*

Abgabe von Cyanid / *Release of Cyanide*

Untersuchungsverfahren / *Test Method*

Migrationsbedingungen / *migration conditions* :
Wasser / *water*, 10 d, 40°C
Bestimmung von Cyanid in Anlehnugn an DIN 38405 D13. / *Determination of cyanide following DIN 38405 D13*

Untersuchungsergebnisse / *Test Results*

Probe / *sample*	CN^-	Bewertung / *Assessment*
Graphit-Folie / *graphite foil*	<0,001	erfüllt / *passed*

Farblässigkeit / *Transfer of Colourants*

Untersuchungsverfahren / *Test Method*

Prüfung in Anlehnung an die Empfehlung B II IX der Kunststoffkommission des BfR. / *Test in dependence on Recommendation B II IX of the BfR*:
Vier Filterpapiere wurden jeweils mit deionisiertem Wasser, 3 %iger Essigsäure, 10 % Ethanol und Erdnußöl getränkt und mit der Probe bei 50° C und einem Druck von 1 kg/dm^2 in Kontakt gebracht. Nach 5 Stunden wurde der Farbübergang auf das Filterpapier bewertet. / *Four filter papers were soaked with either deionized water, 3 % acetic acid, 10 % ethyl alcohol and peanut oil and put in contact with the sample at 50°C and a pressure of 1 kg/dm^2.*

Untersuchungsergebnisse / *Test Results*

	Graphit-Folie / *graphite foil*
Wasser	kein Farbübergang / *no transfer of colourants*
3 %ige Essigsäure	kein Farbübergang / *no transfer of colourants*
10 % Ethanol	kein Farbübergang / *no transfer of colourants*
Erdnußöl	kein Farbübergang / *no transfer of colourants*
Bewertung / *Assessment**	erfüllt / *passed*

*Anforderung nach BfR: kein Transfer von Farbstoffen auf Lebensmittel /
requirement according to BfR: no transfer of colourants to foodstuffs.

Gesamtmigration / *Total Migration*

Untersuchungsverfahren / *Test Method*

Migrationsbedingungen / *migration conditions* :

Wasser / *water*	10	d	40	°C
3 % Essigsäure / *3 % acetic acid*	10	d	40	°C
Isooctan / *isooctane*	2	d	20	°C
95% Ethanol / *95% ethylalcohol*	10	d	40	°C

Es wurde der Abdampfrückstand gravimetrisch durch Eindampfen des Migrats und Trocknen bei 105°C bestimmt. / *The evaporation residue was gravimetrically determined by evaporating the migrate and drying it at 105°C.*

Untersuchungsergebnisse / *Test Results*

Probe / *sample*	Ergebnis / *result* [mg/dm²]	Bewertung / *Assessment**
Wasser / *water*		
Graphit-Folie / *graphite foil*	1,9	erfüllt / *passed*
3 % Essigsäure / *3 % acetic acid*		
Graphit-Folie / *graphite foil*	1,4	erfüllt / *passed*
Isooctan / *isooctane*		
Graphit-Folie / *graphite foil*	< 1,0	erfüllt / *passed*
95% Ethanol / *95% ethylalcohol*		
Graphit-Folie / *graphite foil*	1,4	erfüllt / *passed*

*Grenzwert nach Bedarfsgegenständeverordnung *limit according to „Bedarfsgegenständeverordnung":*
10 mg/dm² bzw. 60 mg/kg /
Anmerkung / *note:*
Ein Bedarfsgegenstand, der den Gesamtmigrationsgrenzwert um höchstens den nachstehenden Analysentoleranzwert überschreitet, ist daher als richtlinienkonform zu betrachten. / *A material or article that exceeds the overall migration limit by an amount not greater than the analytical tolerance mentioned below should therefore be deemed to be in compliance with this Directive.*
Folgende Analysentoleranzen sind festgestellt worden / *The following analytical tolerances have been observed:*

- 20 mg/kg oder 3 mg/dm² bei Migrationsuntersuchungen, bei denen rektifiziertes Olivenöl oder seine Substitute verwendet werden / *20 mg/kg or 3 mg/dm² in migration tests using rectified olive oil or substitutes*
- 12 mg/kg oder 2 mg/dm² in Migrationsuntersuchungen, bei denen die anderen in den Richtlinien 82/711/EWG und 85/572/EWG angegebenen Simulanzlösemittel verwendet werden. / *12 mg/kg or 2 mg/dm² in migration tests using the other simulants referred to in Directives 82/711/EEC and 85/572/EEC.*

Polycyclische aromatische Kohlenwasserstoffe (PAK) / *Polycyclic aromatic Hydrocarbons (PAH)*

Untersuchungsverfahren / *Test Method*

AA-QCPR-307_03: Extraktion mit n-Hexan, GC-MS. / *Extraction with n-hexane, GC-MS.*

Untersuchungsergebnisse / *Test Results*

Parameter / *parameter*	Dimension	Graphit-Folie / *graphite foil*
Naphthalin	mg/kg	<0,1
2-Methylnaphthalin*	mg/kg	<0,1
1-Methylnaphthalin*	mg/kg	<0,1
Acenaphthylen	mg/kg	<0,1
Acenaphthen	mg/kg	<0,1
Fluoren	mg/kg	<0,1
Phenanthren	mg/kg	<0,1
Anthracen	mg/kg	<0,1
Fluoranthen	mg/kg	<0,1
Pyren	mg/kg	<0,1
Chrysen	mg/kg	<0,1
Benzo(a)anthracen	mg/kg	<0,1
Benzo(b)fluoranthen und / *and* Benzo(k)fluoranthen	mg/kg	<0,1
Benzo(a)pyren	mg/kg	<0,1
Dibenzo(a,h)anthracen	mg/kg	<0,1
Indeno(c,d)pyren	mg/kg	<0,1
Benzo(g,h,i)perylen	mg/kg	<0,1

Parameter / *parameter*	Dimension	Graphit-Folie / *graphite foil*
Summe / *sum*	mg/kg	**0,0**

Phthalatweichmacher in Bedarfsgegenständen mit Lebensmittelkontakt / *Phthalate Softeners in Food Contact Materials*

Untersuchungsverfahren / Test Method

Extraktion mit TBME, Bestimmung mittels GC-MS. / *Extraction with TBME, determination by means of GC-MS.*

Untersuchungsergebnisse / Test Results

Parameter / *parameter*	Dimension / *dimension*	Graphit-Folie / *graphite foil*
Diethylhexylphthalat (DEHP)	%	< 0,03
Dibutylphthalat (DBP)	%	< 0,03
Benzylbutylphthalat (BBP)	%	< 0,03
Diisononylphthalat (DINP)	%	< 0,03
Diisodecylphthalat (DIDP)	%	< 0,03
Mischung aus 50% n-Decyl-n-octylphthalat, 25% Di-n-octylphthalat (DNOP) und 25% Di-n-decylphthalat (DNDP) / *mixture of 50% phthalic aced n-decyl n-octyl ester, 25% phthalic acid di-n-decyl ester, 25% phthalic acid di-n- octyl ester*	%	< 0,03
Kategorie / *category*	-	Mehrwegartikel / *repeated use article*
Bewertung / *assessment**	-	erfüllt / *passed*

Grenzwerte gem. 2007/19/EG für die Verwendung als Hilfsagens /
limit according to 2007/19/EC as technical support agent :

DEHP 0,1 %, zulässig als Weichmacher in Mehrwegartikeln für fettfreie Lebensmittel /
permitted as plasticizer in repeated use articles for nonfat foods

DBP 0,05 %, zulässig als Weichmacher in Mehrwegartikeln für fettfreie Lebensmittel/
permitted as plasticizer in repeated use articles for nonfat foods

BBP 0,1 %, zulässig als Weichmacher in Mehrwegartikeln und Einwegartikel für fettfreie Lebensmittel, außer für Säuglingsanfangsnahrung und Folgenahrung gemäß der Richtlinie 91/321/EWG und bei Erzeugnissen gemäß der Richtlinie 96/5/EG /
permitted as plasticizer in repeated use articles and single use articles for nonfat foods, except for infant formulae and follow-on formulae as defined by Directive 91/321/EEC and products according to Directive 96/5/EC

DINP 0,1 %, zulässig als Weichmacher in Mehrwegartikeln und Einwegartikel für fettfreie Lebensmittel, außer für Säuglingsanfangsnahrung und Folgenahrung gemäß der Richtlinie 91/321/EWG und bei Erzeugnissen gemäß der Richtlinie 96/5/EG /
permitted as plasticizer in repeated use articles and single use articles for nonfat foods, except for infant formulae and follow-on formulae as defined by Directive 91/321/EEC and products according to Directive 96/5/EC

DIDP 0,1 %, zulässig als Weichmacher in Mehrwegartikeln und Einwegartikel für fettfreie Lebensmittel, außer für Säuglingsanfangsnahrung und Folgenahrung gemäß der Richtlinie 91/321/EWG und bei Erzeugnissen gemäß der Richtlinie 96/5/EG /
permitted as plasticizer in repeated use articles and single use articles for nonfat foods, except for infant formulae and follow-on formulae as defined by Directive 91/321/EEC and products according to Directive 96/5/EC

für die Mischung aus 50 % n-Decyl-n-octylphthalat, 25 % Di-n-octylphthalat (DNOP) und 25 % Di-n-decylphthalat (DNDP) liegt der SML bei 5 mg/kg bzw. 0,8 mg/dm^2 / *for the mixture of 50 % phthalic aced n-decyl n-octyl ester, 25 % phthalic acid di-n-decyl ester, 25 % phthalic acid di-n- octyl ester the SML is 5 mg/kg respectively 0,8 mg/dm^2*

LGA QualiTest GmbH
Chemische Produktprüfung / *Chemical Product Testing*

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Prüfbericht Nr. /
test report no. 5681826-AT1.doc

Sensorische Prüfung / *Sensory Test*

Untersuchungsverfahren / *Test Method*

Die Durchführung erfolgte nach DIN 10955. / *The test was carried out according to DIN 10955.*
Migrationsbedingungen / *migration conditions* :
Wasser / *water*, 10 d, 40°C
Anschließend erfolgte die Verkostung in Form einer paarweisen Vergleichsprüfung nach DIN EN ISO 5495. / *Afterwards the samples were tasted using a paired comparative test according to DIN EN ISO 5495.*

Untersuchungsergebnisse / *Test Results*

Probe / *sample*	Ergebnis / *result*	Bewertung / *Assessment**
Graphit-Folie / *graphite foil*	0	erfüllt / passed

*Wenn eine Gesamtnote zwischen 0 bis 2,5 erreicht wird, liegt keine sensorische Abweichung vor und die Probe entspricht diesbezüglich den Anforderungen des § 31 Abs. 1 LFGB. / *If the evaluation is between 0 to 2.5 no sensory deviation is indicated. The sample fulfils the requirements of § 31 LFGB.*

Lebensmittelrechtliche Bewertung / *Assessment according to Food Legislation*

Bei dem geprüften Artikel handelt es sich um einen Bedarfsgegenstand im Sinne des § 2 Abs. 6 Nr. 1 Lebensmittel-, Bedarfsgegenstände- und Futtermittelgesetzbuch (LFGB).
Er unterliegt somit den lebensmittelrechtlichen Anforderungen. / *The article submitted to test is a consumer good in the sense of § 2 clause 6 no. 1 German Code for Foodstuffs, Commodities and Feeding Stuffs. Therefore it has to comply with the legal requirements.*

Hinsichtlich der geprüften Parameter entspricht der Artikel den Anforderungen des § 31 LFGB. / *Regarding the tested parameters the tested article complies with the requirements of § 31 LFGB.*

Nürnberg, 30.10.2008

LGA QualiTest GmbH
Chemische Produktprüfung /
Chemical Product Testing

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LGA QualiTest GmbH
Chemische Produktprüfung

Dr. Dorothee Boeck
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Leiterin Fachzentrum /
Head Competence Centre

Sandra Wägner
Staatl. gepr. Lebensmittelchemikerin

Fraunhofer
IVV

Test report, part 1

Determination of metals soluble in hydrochloric acid from a graphite films

Customer:	SGL Carbon GmbH 86400 Meitingen
IVV Order no.:	PA/4148/17
Date of order:	31.03.2017
Sample receipt:	31.03.2017
Testing period:	04.04. – 13.04.2017
Date of report	09.05.2017
Sample storage:	Remaining test material will be stored in the institute for six months.
Total pages of the report:	6

The results relate only to the investigated samples.

1 Scope

The investigated graphite material is intended to be used in food processing machines (repeated use, huge amounts of food) as sealing materials and not in food packaging applications.

In the USA there are no statutory regulations for the composition resp. the purity of graphite (CAS 7782-42-5) in contact with food. However, substances used as components of articles that contact food shall be of a purity suitable for its intended use according to 21 CFR 174.5 "General provisions applicable to indirect food additives".
The restriction of various metals/elements in the graphite film is determined according to the BfR recommendations IX (pigments) and LII (filler). For this purpose the substances soluble in 0.07 N hydrochloric acid were analysed according DIN 53770 part 1.

2 Sample material

The customer provided the following sample material:

Sample: SIGRAFLEX® graphite film F05010Z

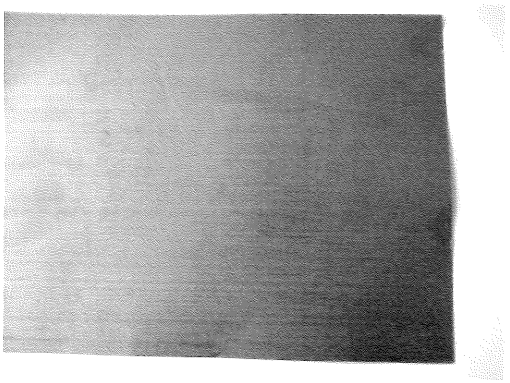

Figure 1: Sample – SIGRAFLEX® graphite film F05010Z

3 Method

Determination of metal content soluble in 0.07 N hydrochloric acid

Extraction and quantification was determined by an external laboratory which is accredited for both methods.

The extraction with 0.07 N hydrochloric acid was performed according to DIN 53770 part 1.

The elements were determined quantitatively according to DIN EN ISO 17294-2 "Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 2: Determination of 62 elements". Rhodium and Rhenium are used as internal standards. Calibration was performed by using multi element standards (simple linear).

4 Results

Table 1: Quantification of element content soluble in 0,07 N hydrochloric acid

Element	Symbol	Soluble content [mg/kg]	Quantification limit [mg/kg]
Aluminium	Al	< 1	1
Arsenium	As	< 0.1	0.1
Barium	Ba	< 0.5	0.5
Cadmium	Cd	< 0.1	0.1
Cerium	Ce	< 0.1	0.1
Cobalt	Co	< 0.1	0.1
Chromium	Cr	< 0.5	0.5
Caesium	Cs	< 0.1	0.1
Copper	Cu	< 0.5	0.5
Iron	Fe	1	1
Gallium	Ga	< 0.1	0.1
Hafnium	Hf	< 0.1	0.1
Mercury	Hg	< 0.02	0.02
Lanthanum	La	< 0.1	0.1
Lithium	Li	< 0.1	0.1

Manganese	Mn	< 1	1
Molybdenum	Mo	< 0.5	0.5
Nickel	Ni	< 0.5	0.5
Lead	Pb	**0.05**	0.05
Rubidium	Rb	< 0.1	0.1
Antimony	Sb	< 0.1	0.1
Selenium	Se	< 0.5	0.5
Tin	Sn	< 0.5	0.5
Strontium	Sr	< 1	1
Tantalum	Ta	< 0.1	0.1
Tellurium	Te	< 0.1	0.1
Thorium	Th	< 0.1	0.1
Titanium	Ti	< 1	1
Uranium	U	< 0.1	0.1
Vanadium	V	< 0.5	0.5
Wolfram	W	< 0.5	0.5
Yttrium	Y	< 0.1	0.1
Zinc	Zn	< 1	1
Zirconium	Zr	< 1	1

5 Food regulatory assessment

In the 0.07 N hydrochloric acid extraction of the investigated sample material iron (Fe) and lead (Pb) were found at the respective quantification limits.

Iron, elemental (CAS 7439-89-6) is defined as ´generally recognized as safe´ (GRAS) according 21 CFR 184.1375 and therefore no limits for the use in food contact materials exist.

Lead is not regulated according the 21 ´Code of Federal Regulations´ (CFR) for the use in food contact materials. For the food regulatory assessment BfR recommendation IX and LII are used.
According to the BfR recommendation IX on pigments and BfR recommendation LII on fillers the lead content soluble in 0.07 N hydrochloric acid must not exceed 0,01 % (100 mg/kg).

The BfR recommendation IX on pigments and BfR recommendation LII on fillers gives the following limits for the content of elements which are soluble in

hydrochloric acid according to DIN 53770 (see Table 2 and Table 3).

Table 2: Restrictions of elements soluble in 0.07 N hydrochloric acid according to BfR recommendation IX

Element	Symbol	Restriction [%]	Restriction [mg/kg]
Lead	Pb	0.01	100
Arsenium	As	0.01	100
Mercury	Hg	0.005	50
Selenium	Se	0.01	100
Barium	Ba	0.01	100
Chromium	Cr	0.1	1000
Cadmium	Cd	0.01	100
Antimony	Sb	0.05	500

Table 3: Restrictions of elements soluble in 0.1 N hydrochloric acid according to BfR recommendation LII

Element	Symbol	Restriction [%]	Restriction [mg/kg]
Lead	Pb	0.01	100
Arsenium	As	0.01	100
Mercury	Hg	0.0005	5
Barium (from Barium sulfate)	Ba	0.01	100
Cadmium	Cd	0.01	100
Antimony	Sb	0.005	50

The quantification limits obtained by the used analytical method are below these restrictions given in the BfR recommendations IX and LII.

Therefore, the investigated graphite film is in compliance with the specifications for metals/elements given in the BfR recommendations IX and LII.

6 Signatures

Fraunhofer Institute
Process Engineering
and Packaging

Dr. Diana Kemmer
(Dep. Head of Migration Laboratory)

Freising. 09.05.2017

Maria Gierl
(Scientist in charge)

Fraunhofer
IVV

Test report, part 2

Determination of polycyclic aromatic hydrocarbons (PAHs) in the material

Customer:	SGL Carbon GmbH 86400 Meitingen
IVV Order no.:	PA/4148/17
Date of order:	31.03.2017
Sample receipt:	31.03.2017
Testing period:	03.04. – 26.06.2017
Date of report	27.07.2017
Sample storage:	Remaining test material will be stored in the institute for six months.
Total pages of the report:	6

The results relate only to the investigated samples.

DAkkS
Deutsche
Akkreditierungsstelle
D-PL-11140-04-00

Fraunhofer-Institut für Verfahrenstechnik und Verpackung, Giggenhauser Str. 35, D-85354 Freising

1 Scope

The investigated graphite material is intended to be used in food processing machines (repeated use, huge amounts of food) as sealing materials and not in food packaging applications.

In USA there are no statutory regulations for the composition resp. the purity of graphite (CAS 7782-42-5) in contact with food. However, substances used as components of articles that contact food shall be of a purity suitable for its intended use according to 21 CFR 174.5 "General provisions applicable to indirect food additives".

High temperature is needed for the production of synthetic graphite. Thereby various polycyclic aromatic hydrocarbons (PAH) can be generated. The US Environmental Protection Agency (EPA) has selected 16 PAH-substances from more than 100 substances and compiled in a list, that are most frequently found in the environment ("EPA-PAH").

These 16 EPA-PAHs were analysed in the investigated graphite film.

2 Sample material

The customer provided the following sample material:

Sample: SIGRAFLEX® graphite film F05010Z

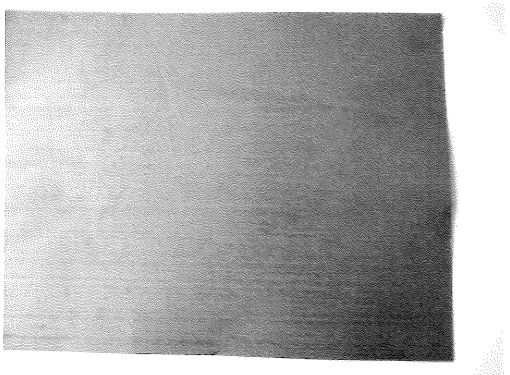

Figure 1: Sample – SIGRAFLEX® graphite film F05010Z

3 Method

Determination of 16 polycyclic aromatic hydrocarbons (PAHs) in the material

Accredited Fraunhofer IVV method PA 1.601

a) Extraction

The sample was ground with a centrifugal mill (Retsch ZM-200) to a particle size of ≤ 250 µm. Approx. 0.5 g of the ground and homogenized sample were spiked with a mixture of isotope labeled PAH-standard (internal standard) solution was extracted under the following extraction conditions:
pressure: 100 bar, temperature: 120 °C, cycles: 3 cycles each 15 min (static)

The sample extracts were reduced to a small volume and measured by GC-MS.

The analysis was performed in triplicate.

b) Quantification of 16 EPA – PAHs

The PAH content of the extraction solutions was determined by a low resolution mass spectrometer (Shimadzu QP 2010 plus) in the single ion monitoring (SIM) mode.

The PAH content in the sample solutions was determined by the ratio of an external calibration and the isotopic labeled standard substances. All values were corrected by blank samples.

4 Results

Polycyclic hydrocarbons (PAH) in the material

Analyt	Concentration in the material [µg/kg]	Area related migration [µg/dm²][1]
Naphthalene	**105** *(d.l.: 10)*	**0.5** *(d.l.: 0.05)*
Acenaphthylen	< d.l. *(d.l.: 10)*	< d.l. *(d.l.: 0.05)*
Acenaphthen	< d.l. *(d.l.: 20)*	< d.l. *(d.l.: 0.1)*
Fluoren	< d.l. *(d.l.: 50)*	< d.l. *(d.l.: 0.3)*
Phenanthren	**39** *(d.l.: 20)*	**0.2** *(d.l.: 0.1)*
Anthracene	< d.l. *(d.l.: 25)*	< d.l. *(d.l.: 0.1)*
Fluoranthene	**34** *(d.l.: 10)*	**0.2** *(d.l.: 0.05)*
Pyrene	< d.l. *(d.l.: 600)*[2]	< d.l. *(d.l.: 2.9)*
Benzo(a)anthracene	< d.l. *(d.l.: 10)*	< d.l. *(d.l.: 0.05)*
Chrysene	**220** *(d.l.: 10)*	**1.1** *(d.l.: 0.1)*
Benzo(b)fluoranthene	< d.l. *(d.l.: 10)*	< d.l. *(d.l.: 0.05)*
Benzo(k)fluoranthene	< d.l. *(d.l.: 10)*	< d.l. *(d.l.: 0.05)*
Benzo(a)pyrene	< d.l. *(d.l.: 5)*	< d.l. *(d.l.: 0.03)*
Indeno(1,2,3-cd)pyrene	< d.l. *(d.l.: 10)*	< d.l. *(d.l.: 0.05)*
Dibenz(a,h)anthracene	< d.l. *(d.l.: 20)*	< d.l. *(d.l.: 0.1)*
Benzo(g,h,i)perylene	< d.l. *(d.l.: 20)*	< d.l. *(d.l.: 0.1)*

d.l. detection limit
[1] calculated with a grammage of approx. 4.9 g/dm²
[2] due to matrix influence the detection limit of pyrene is 600 µg/kg material

5 Food regulatory assessment

The US Environmental Protection Agency (EPA) has selected16 PAH-substances from more than 100 substances and compiled in a list, that are most frequently found in the environment ("EPA-PAH").

In the investigated sealing SIGRAFLEX® material naphthalene, phenanthrene, fluoranthene and chrysene were detected at the detection limit of 10 resp. 20 µg/kg material. Due to strong matrix influence the detection limit of pyrene is 600 µg/kg material.

Naphthalene was quantified to be 105 µg/kg in the material. The oral reference dose in drinking water is 20 µg/kg body weight / day for naphthalene according to US-EPA[1]. For an adult person with 60 kg body weight, this reference dose corresponds to 1200 µg naphthalene (1200 µg/kg food under the assumption that 1 kg food will be consumed per day).
Calculating with the grammage of approx. 4.9 g/dm^2, the amount of 105 µg/kg corresponds to an area related migration potential of 0.5 µg/dm^2 (under assumption of total transfer – worst case scenario). Therefore the maximum possible migration of naphthalene from the investigated graphite film is below the described reference dose for all practicable surface-to-volume ratios for the real application as sealing material in the food industry.

Fluoranthene was quantified to be 34 µg/kg in the material. The oral reference dose of fluoranthene is 40 µg/kg body weight / day according to EPA[2]. For an adult person with 60 kg body weight, this reference dose corresponds to 2400 µg fluoranthene (2400 µg/kg food under the assumption that 1 kg food will be consumed per day).
Calculating with the grammage of approx. 4.9 g/dm^2, the amount of 34 µg/kg corresponds to an area related migration potential of 0.2 µg/dm^2 (under assumption of total transfer – worst case scenario). Therefore the maximum possible migration of fluoranthene from the investigated graphite film is below the described reference dose for all practicable surface-to-volume ratios for the real application as sealing material in the food industry.

Chrysene is classified as a possible human carcinogenic substance[1,3]. Until now an oral reference dose for drinking water is not set by the EPA.
Until now phenanthrene is not classified as a human carcinogenic substance ("classification – D, not classifiable as to human carcinogenicity")[1,4].

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[1] 2012 Edition of the Drinking Water Standards and Health Advisories, EPA 822-S-12-001, Office of Water U.S. Environmental Protection Agency Washington, DC, Date of update: April, 2012
[2] Integrated Risk Information System (IRIS) U.S. Environmental Protection Agency Chemical Assessment Summary, STATUS OF DATA for Fluoranthene
[3] Integrated Risk Information System (IRIS) U.S. Environmental Protection Agency Chemical Assessment Summary, STATUS OF DATA for Chrysen
[4] Integrated Risk Information System (IRIS) U.S. Environmental Protection Agency Chemical Assessment Summary, STATUS OF DATA for Phenanthrene

Many polycyclic aromatic hydrocarbons (PAHs) are classified as human carcinogenic substances. Thereby benzo(a)pyrene is used as an indicator substance for the PAHs substances because, beside its carcinogenic property benzo(a)pyrene is almost certainly a mutagenic and reprotoxic substance[5]. According to the EPA a maximum amount of benzo(a)pyrene of 0.0002 mg/liter (0.2 µg/liter) in the drinking water is acceptable. For the assessment of phenanthrene and chrysene the described limit of benzo(a)pyrene is used. Phenanthrene was quantified to be 39 µg/kg in the graphite film. Calculating with the grammage of approx. 4.9 g/dm² the amount corresponds to an area related migration potential of 0.2 µg/dm² (under assumption of total transfer – worst case scenario). Up to a surface-to-volume ratio of maximum 1 dm²/kg food the maximum possible migration of phenanthrene from the investigated graphite film meets the described limit.
Chrysene was quantified to be 220 µg/kg in the graphite film. Calculating with the grammage of approx. 4.9 g/dm² the amount corresponds to an area related migration potential of 1.1 µg/dm² (under assumption of total transfer – worst case scenario). Up to a surface-to-volume ratio of maximum 0.18 dm²/kg food the maximum possible migration of chrysene from the investigated graphite film meets the described limit.

For pyrene an area-related migration potential of 2.9 µg/dm², assuming a grammage of 4.9 g/dm², was determined. For pyrene an oral reference dose of 30 µg/kg body weight / day for drinking water is laid down by EPA (2012 Edition of the Drinking Water Standards and Health Advisories[6]). For an adult person with 60 kg body weight, this reference dose corresponds to 1800 µg pyrene (1800 µg/kg food under the assumption that 1 kg food will be consumed per day). Therefore the maximum possible migration of pyrene from the investigated graphite film is below the described reference dose for all practicable surface-to-volume ratios for the real application as sealing material in the food industry.

6 Signatures

Fraunhofer Institute
Process Engineering
and Packaging

Freising. 27.07.2017

M. Gierl

Maria Gierl
(Scientist in Charge)

Ludwig Gruber
(Head of Contaminants Laboratory)

[5] Bundesinstitut für Risikobewertung (BfR), Polyzyklische aromatische Kohlenwasserstoffe (PAK) in Spielzeug, 2009
[6] 2012 Edition of the Drinking Water Standards and Health Advisories, EPA 822-S-12-001, Office of Water U.S. Environmental Protection Agency Washington, DC, Date of update: April, 2012

Fraunhofer
IVV

Test report, part 3

Screening analysis of a graphite film

Customer:	SGL Carbon GmbH 86400 Meitingen
IVV Order no.:	PA/4148/17
Date of order:	30.03.2017
Sample receipt:	30.03.2017
Testing period:	04.04. – 26.04.2017
Date of report:	28.07.2017
Sample storage:	Remaining test material will be stored in the institute for six months.
Total pages of the report:	7

The results relate only to the investigated samples.

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D-PL-11140-04-00

1 Scope

The investigated graphite material is intended to be used in food processing machines (repeated use, huge amounts of food) as sealing materials and not in food packaging applications.

In USA there are no statutory regulations for the composition resp. the purity of graphite (CAS 7782-42-5) in contact with food. However, substances used as components of articles that contact food shall be of a purity suitable for its intended use according to 21 CFR 174.5 "General provisions applicable to indirect food additives".
The extractions of the graphite film are tested with respect to possible migratable organic contaminations by screening analysis.

2 Sample material

The customer provided the following sample material:

Sample: SIGRAFLEX® graphite film F05010Z

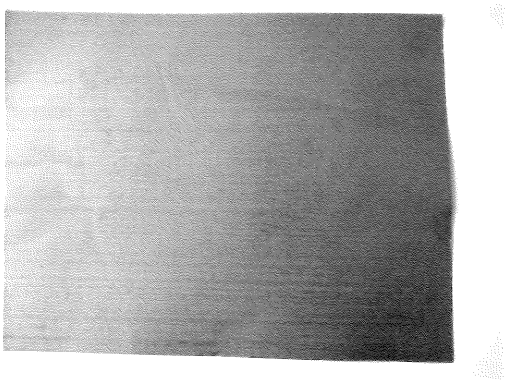

Figure 1: Sample – SIGRAFLEX® graphite film F05010Z

3 Methods

Screening analysis of migratable compounds by extraction

Accredited Fraunhofer IVV method 1.337

0.5 dm^2 of the sample (corresponding to 2.5 g film) cut in small pieces were extracted with 10 ml dichloromethane (DCM) for 3 days at 40 °C resp. with 10 ml 95 % ethanol for 3 days at 60 °C by total immersion (in duplicate). An internal standard of butylated hydroxyanisole (BHA) und Tinuvin 234 was added to an aliquot of the extracts, and analysed by gas chromatography with flame

ionisation detection (GC-FID) for semi-volatile compounds. To enhance the detection limit the internal standard was added to the rest of the extraction solutions which were then reduced to approx. 1 ml under a nitrogen stream and then analysed by GC-FID. Peaks of interest were semi-quantified using the internal standard BHA.

The extraction solutions were analysed by gas chromatography with flame ionisation detection (GC-FID): DB-1-capillary column (length 30 m, inner diameter 0.25 mm, film thickness 0.25 µm) and the following temperature programme: 50 °C (2 min isotherm) up to 340 °C with a heating rate of 10 °C/min, then 10 min isotherm at 340 °C.

With this method organic components with molecular range of 150 – 700 Dalton will be determined.

The identification of the main compounds was done by GC analysis coupled with mass spectrometry. GC/MS-System: ThermoFinnigan SSQ, column: Optima-5-MS - 30 m length - 0.25 mm i.d. - 0.25 µm film thickness, temperature programme: 50 °C (2 min), heating rate 10 °C min^{-1}, 340 °C (30 min), full scan mode, mass range *m/z* 40 - 800.
The identification of the spectra was done by comparison with the NIST spectra library. A confirmation of the suggested spectra by analysis of a respective standard was not done.

4 Results

The gas chromatograms of the extraction solutions are displayed in Figure 2 to Figure 5. Characteristic peaks in the extracts were defined as fingerprint components and semi-quantified using the internal standard BHA (Table 1).

The detected peaks were too small on identification using GC-MS analysis.

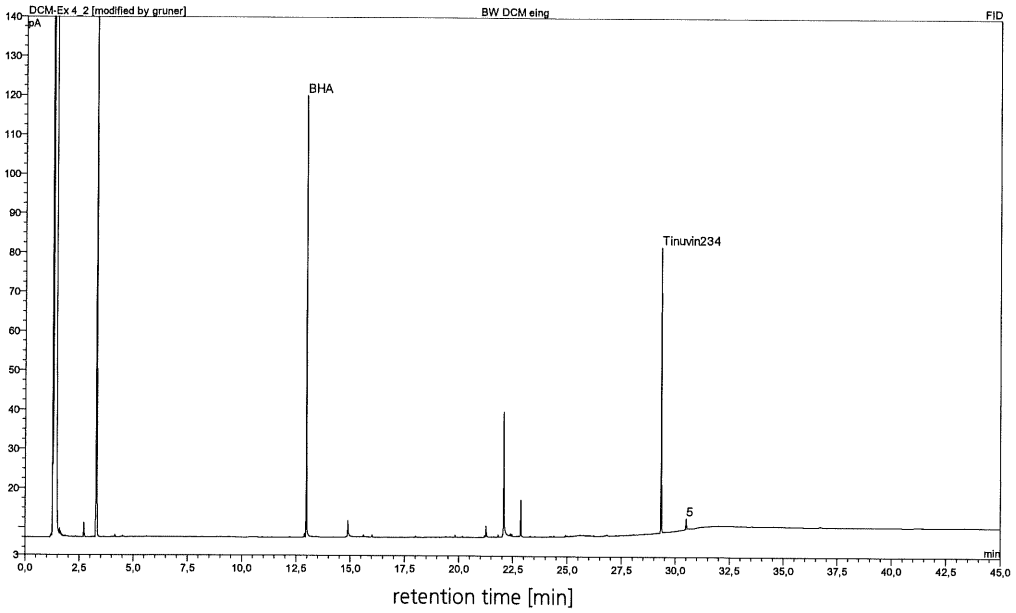

Figure 2: Gas chromatogram of the DCM solvent blank

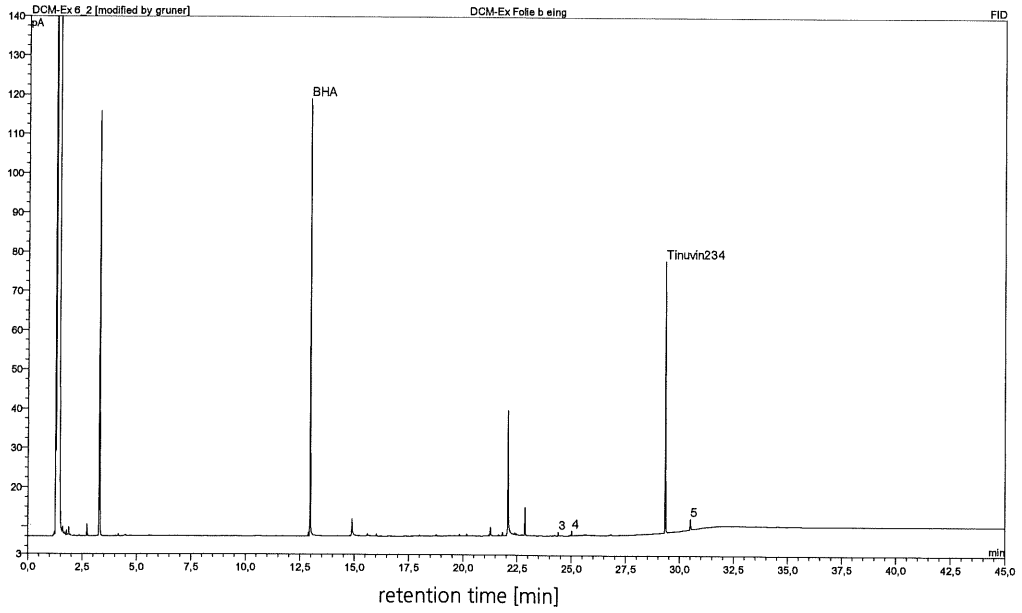

Figure 3: Gas chromatogram of the DCM extract of the sample

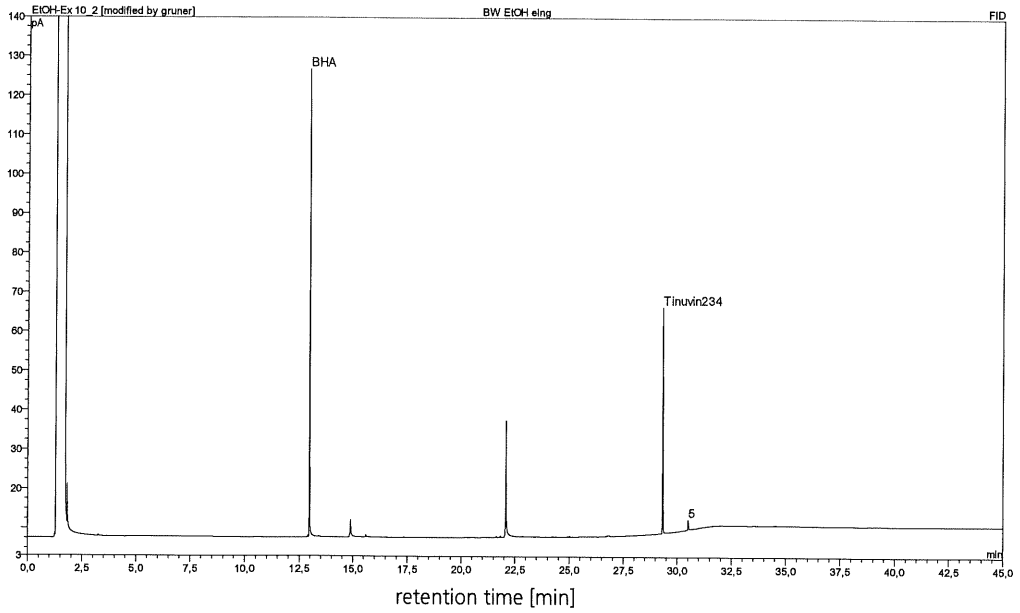

Figure 4: Gas chromatogram of the 95 % ethanol solvent blank

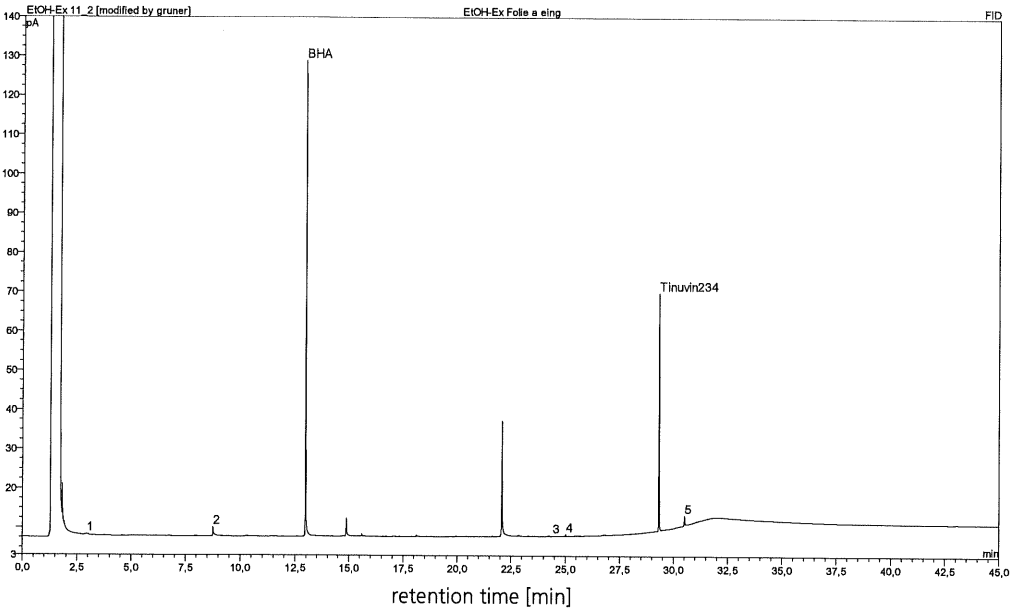

Figure 5: Gas chromatogram of the 95 % ethanol extract of the sample

Table 1 Semi-quantification of fingerprint components in extraction solutions, results given in µg/dm²

	DCM extraction solution [µg/dm²]		95 % ethanol extraction solution [µg/dm²]	
Peak	solvent blank	sample	solvent blank	sample
1	< dl	< dl	< dl	< dl
2	< dl	< dl	< dl	5.4
3	< dl	< dl	< dl	< dl
4	< dl	< dl	0.4	0.3
5	3.8	4.1	3.8	3.1

The detection limit (dl): 0.2 µg/dm²

5 Food regulatory assessment

The evaluation of the applicablility of the graphite film for food processing machines and equipments is based on the possible migration of components from the graphite film into food and the resulting maximum concentrations in food.

With the performed screening analysis of the dichloromethane and 95 % ethanol extraction solutions possible migratable organic contaminants were determined. By comparison with the blank solvents only one not-identified peak (peak 2) in the 95 % ethanol extraction solution was detected. For this peak an area related extraction value of 5.4 µg/dm² results from the semi-quantification.

For the evaluation of the possible mass transfer the Threshold of Regulation – concept (TOR) of the US-FDA is used. On the basis of a statistic evaluation of the toxicological no-effect-level resp. of the tolerable intake of assessed substances, FDA assessed a daily intake of 1.5 µg of a substance as negligibly small, so that no regulation by the authorities for this substance is deemed necessary (21 CFR 170.39). Thereby the derived exposition according FDA results from a daily food intake of 3 kg (including beverages) and the statistic fraction of the assessed material in food contact in relation of all materials in food contact (consumption factor CF).

No statistical data for the use of graphite film in direct food contact was available to us. When the fraction is low and no statistical data are available, a consumption factor of 0.05 is used for the calculation. This corresponds to a maximum migration of 10 µg/kg (10 ppb).

According to the client's information various sizes of the graphite film will be used in contact with food. The above described limit of 10 µg/kg will be met if at least 450 g food will be in contact with 1 dm² contact surface area of the graphite film.

6 Signatures

Fraunhofer Institute
Process Engineering
and Packaging

Freising, 28.07.2017

Maria Gierl
(Scientist in Charge)

Anita Gruner
(Technician)