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Aschaffenburg, 11 December 2020

From:

Be-pf

Authorized by: Behrendt

## REPORT

Order No.:

13934/41

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pages

Client:

Progroup Board GmbH

Horstring 12 76829 Landau

Germany

Date of order:

12 August 2020

Receipt of sample material:

14 August 2020

Origin of sample material:

Geschäftsführer: Dr. Ralph Derra . Handelsregister Aschaffenburg HRB 3329

From the client

Purpose:

Analysis of corrugated board grades for their compliance

with the demands on food contact materials

(Dr. Derra) Managing Director

Officially certified and authorized food chemist

(Behrendt)

The present report refers exclusively to the samples as laid out therein. Information and statistical data on the results can be obtained on request.

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## Sample Material

For analysis the following sample material was in hand:

Sample 1:	1.02B N2
Sample 2:	1.03B W1
Sample 3:	1.10B N2
Sample 4:	1.22C X1
Sample 5:	1.24B Q7
Sample 6:	1.25E
Sample 7:	1.26B C2
Sample 8:	1.27E L2
Sample 9:	1.36B C2
Sample 10:	1.55B X6
Sample 11:	2.35BE
Sample 12:	2.50BC N2
Sample 13:	2.71BC N1
Sample 14:	2.90 BC
Sample 15:	2.92 AC
Sample 16:	3.90 AAC
Sample 17:	3.91 AAC
Sample 18:	3.92 AAC
Sample 19:	3.95 AAC
Sample 20:	3.96 AAC
2	

Unless stated differently, the samples 1 - 10 were examined as **mixed sample 1** and the samples 11 - 20 were examined as **mixed sample 2**.

## **Carrying out of the Tests**

Examination period: 22 October 2020 to 26 November 2020

## 1. Determination of the Grammage \*

The determination was performed according to DIN EN ISO 536:2012-11 after conditioning of the sample at 23 °C/50 % relative humidity which is prescribed as standard atmosphere with a reduced amount of test specimens.

#### Result:

Mixed sample 1: 392 g/m<sup>2</sup>  $\triangleq$  367 g dry matter/m<sup>2</sup> Mixed sample 2: 1155 g/m<sup>2</sup>  $\triangleq$  1077 g dry matter/m<sup>2</sup>

## 2. Determination of the Moisture Content \*

The determination was performed as single determination according to DIN EN ISO 638:2009-01 in the condition as received.

#### Result:

Mixed sample 1: 6.1 % Mixed sample 2: 6.4 %

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# 3. Preparation of Extracts \*

The extracts were prepared according to the "Methodensammlung zur Untersuchung von Papier, Karton und Pappe für den Lebensmittelkontakt" (collection of methods for the examination of paper and board for food contact) of the BfR as well as according to DIN EN 645:1994-01, 647:1994-01 and 15519:2008-01. The selection of suitable procedures for simulating the transfer of substances was performed according to the corresponding BfR guideline ("Leitfaden zur Überprüfung der Stoffübergänge von Bedarfsgegenständen aus Papier, Karton und Pappe").

Water:

24 hours at 23 °C

## 4. Determination of Methanal (Formaldehyde) in the Water Extract \*

The determination was performed according to DIN EN 1541:2001-07 photometrically in line with the acetylacetone method.

Result:

Mixed sample 1 + 2: not quantifiable < 0.004

mg/g

# 5. Determination of Pentachlorophenol (PCP) in the Water Extract \*

The determination was performed according to DIN EN ISO 15320:2011-08 by means of GC-ECD after concentration at a column and esterification.

Result:

Mixed sample 1:

0.017

mg/kg dry matter

Mixed sample 2:

0.018

mg/kg dry matter

## 6. Determination of the Transfer of Antimicrobial Constituents \*

The determination was made according to DIN EN 1104:2019-01. Test specimens of a diameter of 10 mm were placed onto an inoculated nutrient medium and then incubated. The inhibition zone is indicated as total diameter (including the test specimen).

Result:

with Aspergillus niger:

Sample 1 - 20:

Microbial growth up to the edges of the test specimens, no inhibition zone.

with Bacillus subtilis:

Sample 1 - 6:

Microbial growth up to the edges of the test specimens. Presence of a modification of the test microorganism B. subtilis growth at the edges of the test pieces. Presence of a microbial contaminant of < 2 mm around the test pieces.

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Sample 7 - 20:

Microbial growth up to the edges of the test specimens. Presence of a

microbial contaminant of < 2 mm around the test pieces.

#### Comment:

According to the current state of standardization, proof of the presence of an inhibition zone is provided by the absence of test microorganism growth in a minimum diameter of 14 mm. Therefore, a transfer of antimicrobial constituents is considered as not detected.

# 7. <u>Determination of the Specific Migration into Tenax® (Modified Polyphenylene Oxide)</u>\*

The migration was performed according to DIN EN 14338:2004-03.

Conditions A:

10 days at 40 °C

Conditions B:

30 days at 40 °C

Testing procedure:

one-sided contact

Subsequently, the volatile components adsorbed onto Tenax were extracted.

## 7.1. Gas chromatographic Analysis

The determination was performed according to SOP 160.200 by means of GCMS after extraction with methyl *tert*-butylether.

#### a) Sum of the volatile components

The volatile components were summarized semi-quantitatively using deuterated nonadecane as internal standard.

Result:

Conditions A:

Sample 7 + 10: Sample 19 + 20:

1.5

mg/dm<sup>2</sup>

1.2

mg/dm<sup>2</sup>

#### b) Specific Evaluation

In addition, an examination for the below listed contaminants was performed.

Result:

Conditions A:

Sample 7 + 10 + 19 + 20:

Diisopropylnaphthalene (DIPN) Other solvent	[38640-62-9]	not quantifiable not quantifiable	0.05 0.05	mg/dm² mg/dm²
Benzophenone 4-Methyl benzophenone	[119-61-9] [134-84-9]	not quantifiable not quantifiable	0.02 0.02	mg/dm² mg/dm²

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Diisobutyl phthalate	[84-69-5]	not quantifiable	<	0.02	mg/dm²
Dibutyl phthalate	[84-74-2]	not quantifiable	<	0.02	mg/dm²
Di(2-ethylhexyl) phthalate	[117-81-7]	not quantifiable	<	0.05	mg/dm²
Di-(2-ethylhexyl) adipate 2,2,4-trimethyl-1,3-pentanediol	[103-23-1]	not quantifiable	<	0.05	mg/dm²
diisobutyrate (TXIB) Diethylene glycol dibenzoat	[6846-50-0]	not quantifiable	<	0.02	mg/dm²
	[120-55-8]	not quantifiable	<	0.002	mg/dm²
Benzyl-2-naphthylether	[613-62-7]	not quantifiable	<	0.002	mg/dm²

#### 7.2. Mineral Oil (MOSH/MOAH)

The determination of the paraffinic, naphthenic mineral oil hydrocarbons (MOSH) and of the aromatic mineral oil hydrocarbons (MOAH) was performed according to the method published by the German "National Reference Laboratory for Materials in contact with food".

After extraction with hexane, the analysis was performed by means of on-line coupled HPLC-GC-FID using internal standards. In both fractions the chromatographically not resolved hump including signals on top was integrated. Hydrocarbon compounds not defined as mineral oil were deducted during the quantification.

#### Conditions B:

Result: MOSH			M			
	< C <sub>16</sub>	$C_{16} - < C_{35}$	$C_{20} - \le C_{35}$	< C <sub>16</sub>	$C_{16} - \le C_{35}$	
Sample 7:	< 0.08	0.88	0.54	< 0.08	0.11	mg/dm²
Sample 10:	< 0.08	0.95	0.55	< 0.08	0.10	mg/dm²
Sample 19:	< 0.08	0.90	0.50	< 0.08	0.09	mg/dm²
Sample 20:	< 0.08	0.71	0.44	< 0.08	0.05	mg/dm²

# 8. Determination of the Heavy Metals in Packagings \*

The determination was performed after microwave disintegration by means of AAS or ICP-OES. It applies to those metals which are restricted according to the European Packaging Directive 94/62/EC as well as to the US American CONEG legislation.

#### Result:

#### Mixed sample 1:

(Pb):			5.3	mg/kg
(Cd):	not determinable	<	0.5	mg/kg
(Hg):	not determinable	<	0.25	mg/kg
(Cr):			3.7	mg/kg
(Be):	not determinable	<	10	mg/kg
	(Hg): (Cr):	(Cd): not determinable (Hg): not determinable (Cr):	(Cd): not determinable < (Hg): not determinable < (Cr):	(Cd): not determinable < 0.5 (Hg): not determinable < 0.25 (Cr): 3.7

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#### Mixed sample 2:

Lead	(Pb):			5.1	mg/kg
Cadmium	(Cd):	not determinable	<	0.5	mg/kg
Mercury	(Hg):	not determinable	<	0.25	mg/kg
Chromium	(Cr):			3.6	mg/kg
Beryllium	(Be):	not determinable	<	10	mg/kg

Limit value 100 mg/kg (sum of Pb, Cd, Hg and Cr(VI)).

<u>Comment</u>: Under the disintegration conditions the total content of chromium including chromium(VI)

is detected.

# 9. Determination of Vinylchloride [75-01-4] \*

The determination was performed according to SOP 160.200 by means of Headspace-GCMS after dissolving the sample in N,N-dimethylacetamide.

Result:

Mixed sample 1 + 2: not quantifiable < 0.5 mg/kg

# 10. Determination of Vinylidene Chloride [75-35-4] \*

The determination was performed according to SOP 160.200 by means of Headspace-GCMS.

Result:

Mixed sample 1 + 2: not quantifiable < 0.5 mg/kg Mixed sample 1 + 2: not quantifiable < 0.5 mg/kg

# 11. <u>Determination of Bisphenol A [80-05-7] and Bisphenol S [80-09-1] in the Water</u> Extract \*

The determination was performed according to SOP 162.200 by means of HPLC-fluorescence or HPLC-UV.

Result:

Mixed sample 1:

Bisphenol A	1.0	mg/kg
Bisphenol S	2.5	mg/kg

Mixed sample 2:

Bisphenol A	0.81	mg/kg
Bisphenol S	2.5	mg/kg

## 12. Determination of o-phenyl phenol [90-43-7] in the Water Extract \*

The determination was performed according to SOP 162.200 by means of HPLC and UV detection.

Result:

Mixed sample 1 + 2: not quantifiable  $< 1.3^{1}$ 

mg/kg

## 13. Determination of Perfluorooctanoic Acid [335-67-1] and Perfluorooctane Sulfonate [2795-39-3] \*

The determination was performed according to SOP 162.200 by means of LCMS in a methanol extract.

Result:

Mixed sample 1 + 2:

Perfluorooctanoic Acid (PFOA): not quantifiable < 0.02 mg/kg

Perfluorooctane Sulfonate (PFOS):

not quantifiable < 0.02

mg/kg

## 14. Determination of Phthalates \*

The determination was performed according to SOP 160.200 by means of GCMS in an acetone extract. The following compounds were considered:

Diisobutyl phthalate	(DIBP)	[84-69-5]
Dibutyl phthalate	(DBP)	[84-74-2]
Di(2-ethylhexyl) phthalate	(DEHP)	[117-81-7]
Di-n-octyl phthalate	(DOP)	[117-84-0]
Benzylbutyl phthalate	(BBP)	[85-68-7]
Dimethylphthalate	(DMP)	[131-11-3]

Limit of quantitation: 1 mg/kg dry matter

Result:

Mixed sample 1:

Diisobutyl phthalate	3.8	mg/kg dry matter
Dibutyl phthalate	3.9	mg/kg dry matter
Di(2-ethylhexyl) phthalate	11	mg/kg dry matter

Mixed sample 2:

Diisobutyl phthalate	2.4	mg/kg dry matter
Dibutyl phthalate	2.9	mg/kg dry matter
Di(2-ethylhexyl) phthalate	9.4	mg/kg dry matter

The remaining compounds were not quantifiable.

<sup>1)</sup> The determination limit had to be raised due to matrix interferences.

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# 15. Determination of the pH value \*

The determination was performed according to ISO 6588 from a cold water extract.

#### Result:

Mixed sample 1:

8.1

Mixed sample 2:

8.0

# 16. Determination of Phosphates \*

The determination was performed according to SOP 160.200 by gas chromatography in a methanol extract.

#### Result:

#### Mixed sample 1 + 2:

Tri-isobutylphosphate	not quantifiable	<	5	mg/kg dry matter
Tributylphosphate	not quantifiable	<	5	mg/kg dry matter
Triphenylphosphate	not quantifiable	<	5	mg/kg dry matter
Diphenyloctylphosphate	not quantifiable	<	5	mg/kg dry matter
Tri(ethylhexyl)phosphate	not quantifiable	<	5	mg/kg dry matter

# 17. Determination of Polycyclic Aromatic Hydrocarbons (PAH) \*

The determination was performed according to the draft standard by means of GCMS. The following compounds were considered:

Naphthalene	[91-20-3]	Benzo[b]naphtho[1,2-d]thiophene	[205-43-6]
2-Methyl naphthalene	[91-57-6]	Benzo[a]anthracene	[56-55-3]
1-Methyl naphthalene	[90-12-0]	Triphenylene/Chrysene [217-59-4	]/[218-01-9]
Acenaphthylene	[208-96-8]	Benzo[b]fluoranthene	[205-99-2]
Acenaphthene	[83-32-9]	Benzo[k]fluoranthene	[207-08-9]
Fluorene	[86-73-7]	Benzo[e]pyrene	[192-97-2]
Phenanthrene	[85-01-8]	Benzo[a]pyrene	[50-32-8]
Anthracene	[120-12-7]	Perylene	[198-55-0]
2-Methyi phenanthrene	[2531-84-2]	Indeno[1,2,3-cd]pyrene	[193-39-5]
Fluoranthene	[206-44-0]	Dibenzo[a,h]anthracene	[53-70-3]
Pyrene	[129-00-0]	Benzo[g,h,i]perylene	[191-24-2]
Benzo[c]phenanthrene	[195-19-7]		000:

## Limits of quantitation:

Acenapthylene, Fluorene, Fluoranthene, Triphenylene/Chrysene 0.03 mg/kg dry matter; all other compounds 0.02 mg/kg dry matter.

#### Result:

#### Mixed sample 1:

Phenanthrene	0.041	mg/kg dry matter
Pyrene	0.034	mg/kg dry matter

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Hexabromo biphenyl (HxBB)

Octabromo biphenyl (OBB)

Decabromo biphenyl (DBB)

Mixed sample 2:

Phenanthrene

0.027

mg/kg dry matter

Pyrene

0.022

mg/kg dry matter

Further compounds listed above were not quantifiable.

# 18. Determination of Polybromated Diphenylethers (PBDE) and Polybromated Biphenyls (PBB)

The determination was performed in collaboration with ARGUK-Umweltlabor GmbH, Oberursel/ Germany by means of GC-ECD or GCMS in an acetone extract. The following compounds were considered:

Tetrabromo diphenylether (TeBDE)

Pentabromo diphenylether (Σ PeBDE – 85, 99, 100)

Hexabromo diphenylether (HxBDE)

Heptabromo diphenylether (HeBDE)

Octabromo diphenylether (Σ OBDE – 196, 197, 203)

Nonabromo diphenylether (NBDE)

Decabromo diphenylether (DBDE)

Limit of quantitation:

PBDE 10 mg/kg; PBE 1 mg/kg

Result:

Mixed sample 1 + 2:

None of the above-listed compounds were quantifiable.

The accreditation applies to the methods marked with \* in the test report (Register no. D-PL-14160-01-01 and D-PL-14160-01-02).

End of report