

Analysis Report

REPORT NUMBER:
933850.1



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INSTITUTE**

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Encl.: 0
Init.: SRV/ULC

Assignor: Imdex A/S
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Item: Analysis of rubber granulate according to DIN 18035-7:2019-12

Sampling: The assignor

Period: Samples received: 30 June 2020
Test performed: 30 June - 26 August 2020

Storage: The test material will be destroyed after 3 months, unless otherwise agreed in writing.

Remark: The account of the method(s) used only concerns the analysed sample(s).

Terms: This test was conducted in accordance with international requirements (ISO/IEC 17025:2017) and in accordance with the General Terms and Conditions of Danish Technological Institute. The test results solely apply to the tested item(s) or to the sub-sample(s) selected for analysis. This analysis report may be quoted in extract only if Danish Technological Institute has granted its written consent.

Date/place: 26 August 2020
Danish Technological Institute, Aarhus
Laboratory for Chemistry and Microbiology

Signature: Søren Ryom Villadsen
Senior Consultant

Label

Sample Number	Sample Name
933850-1	Rubber granulate

Analysis programme

A migration was performed on the sample by transferring approximately 100g of sample into a fitting containing and filling it with approx. 1000 mL of deionized water. The migration was performed at room temperature (approx. 23°C) and samples were mechanically shaken during the migration. The duration of the migration was 24 hours. Subsequently the migration liquid was analysed for total organic content (TOC), conductance, pH, Zn, Pb, Cd, Cr, Zn, Sn, Hg and Cr VI. Migration and analyses were performed by double determination.

There was performed a soxhlet extraction in dichloromethane on the rubber granulate, and the extract was analysed for polyaromatic hydrocarbons (PAH), chlorinated paraffins (SCCP and MCCP) and phthalates.

Results for analysis of migration liquids

Parameter	Method	24 Hour Result	Limit* 24h	Unit	Uncertainty [%]
pH-value	UA-216	7.6	-	-	5
Conductivity at 22°C	UA-217	21	-	µS/cm	5
Dissolved organic compounds (DOC)	UA-205	20	100	mg/l	10
Lead, Pb	ICP/MS	< 0.005	0.025	mg/l	10
Cadmium, Cd	ICP/MS	< 0.005	0.005	mg/l	10
Chromium, Cr	ICP/MS	< 0.005	0.05	mg/l	10
Chromate, Cr(VI) [#]	Spectro-photometry	< 0.005	0.008	mg/l	10
Mercury, Hg	ICP/MS	< 0.001	0.001	mg/l	10
Zinc, Zn	ICP/MS	0.074	0.5	mg/l	10
Tin, Sn	ICP/MS	< 0.005	-	mg/l	10

* Limit according to DIN 18035:2014-10, Table B.1 or requirement from Danish Environment Protection Agency

[#] Determined via total chromium content

UA methods are internal methods at Danish Technological Institute

Results for content analysis of rubber

Parameter	CAS-number	Method	Result [mg/kg]	Limit* [mg/kg]	Uncertainty [%]
EOX**	-	DIN 38414-S17	< 1	100	-
Short chained chlorinated paraffins, C10-C13 (SCCP)	-	Soxhlet + GC/MS	<1000	-	10
Medium chained chlorinated paraffins C14-C17 (MCCP)	-	Soxhlet + GC/MS	<1000	-	10
Benzo[a]pyren (BaP) + Benzo[e]pyren (BeP) ***	50-32-8 + 192-97-2	Soxhlet + GC/MS	0.61	3 + 3	10
Benzo[a]anthracen (BaA)	56-55-3	Soxhlet + GC/MS	< 0.1	3	10
Chrysen (CHR)	218-01-9	Soxhlet + GC/MS	0.13	3	10
Benzo[b]fluoranthen (BbFA)	205-99-2	Soxhlet + GC/MS	< 0.1	3	10
Benzo[j]fluoranthen (BjFA)	205-82-3	Soxhlet + GC/MS		3	10
Benzo[k]fluoranthen (BkFA)	207-08-9	Soxhlet + GC/MS		3	10
Dibenzo[a,h]anthracen (DBAhA)	253-70-3	Soxhlet + GC/MS	< 0.1	3	10
Sum of analysed PAH	-	Soxhlet + GC/MS	0.74	-	-
Bis(2-ethylhexyl) phthalate (DEHP)	117-81-7	DS/ISO 16181 + GC/MS	11	1000	20
Dibutylphthalate (DBP)	84-74-2	DS/ISO 16181 + GC/MS	51	1000	20
Benzylbutylphthalate (BBP)	85-68-7	DS/ISO 16181 + GC/MS	< 5	1000	20
Sum of phthalates	-	DS/ISO 16181 + GC/MS	62	-	-

< Means below the limit of detection.

* Limit according to DIN 18035:2014-10, Table B.1 or requirement from Danish Environment Protection Agency

** Performed by a subcontractor approved by Technological Institute which is under accreditation DAKS D-PL-14081-01-00.

*** It was not possible to separate the compounds in the analysis. The compounds have been reported as a sum of both compounds.

N/A: The result is not available in time for this draft.

Remarks

The product passes for all parameters given in DIN 18035-7:2019-12.

Analysis methods

pH-measurement: The measurement was performed potentiometrically with a pH meter combined with glass electrode.

Detection limit: Not available

Estimated uncertainty: 10% RSD

Conductance: The measurement was performed using an electric conductivity meter

Detection limit: 1 mS/m or 10 μ S/cm

Estimated uncertainty: 10% RSD

DOC determination: DOC was determined as TOC dissolved in the migration liquid using a Shimadzu TOC analyser.

Detection limit: 0.06 mg/l

Estimated uncertainty: 10% RSD

Phthalate determination: A subsample of the sample was extracted in dichloromethane using Soxhlet and subsequently added internal standards of DBP-d₄ and DEHP-d₄. Analyses of the extracts were performed by capillary gas chromatography combined with mass spectrometry (GC/MS). Reference method DS/ISO 16181.

The method used for determination of phthalates does not follow the requirement in DIN 18035-7:2014, see under deviations.

Detection limit: 5 – 20 mg/kg

Estimated uncertainty: 20% RSD

PAH determination: A subsample of the sample was extracted in dichloromethane using Soxhlet. Analyses of the extracts were performed by capillary gas chromatography combined with mass spectrometry (GC/MS) as defined in DIN 18035-7:2019-12.

Detection limit: 0.1 mg/kg

Estimated uncertainty: 15% RSD

SCCP and MCCP determination: A subsample of the sample was extracted in dichloromethane using Soxhlet. Analyses of the extracts were performed by capillary gas chromatography combined with mass spectrometry (GC/MS) as defined in DIN 18035-7:2019-12.

Detection limit: 1000 mg/kg

Estimated uncertainty: 20% RSD

Analysis of metals: The liquid was analysed for the content of lead and cadmium using ICP-MS with CCT in KED-mode using He as collision gas, and 102Rh and 185Re as internal standards. The quantification was performed using traceable, external reference elements. The calibration was verified with independent, traceable control solutions.

The detection limit is 0.06 - 1 μ g/l

Estimated uncertainty: 10 – 15% RSD

Deviations

The product was not extracted with toluene as defined in DIN 18035-7:2019-12. Dichloromethane was used instead.