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## **Analysis Report No. 682507**

**Assignment:** Analyses of content of metals, Phenols, Phthalates, and PCB in samples of rubber granulate.

**Sampling by:** The client

**Sample(s) received:** 30 March 2016

**Test performed:** April to May 2016

**Test results:** The results of the analysis and the method(s) used concern only the sample(s) analysed or the sub-sample(s) selected for analysis.

This analysis has been carried out in accordance with Danish Technological Institute's General Terms and Conditions regarding Commissioned Work Accepted by Danish Technological Institute and in accordance with the current guidelines laid down by DANAK. This analysis report may be quoted in extract only if the Laboratory for Chemistry and Microbiology has approved the extract in writing.

The Laboratory for Chemistry and Microbiology

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## Introduction

The 5 samples of rubber granulate were analysed for content of specific compounds and groups of compounds ordered by Nordisk Kjemikaliegruppe.

## Purpose

The purpose was to determine that the rubber granulates does not contain any critical substances.

## Sample list

Lab. label	Description of the samples
682507-1	IS-01 (Egilshöllin)
682507-2	IS-02 (KR völlum)
682507-3	IS-03 (KA völlum)
682507-4	IS-04 (Varmárskólavöllum)
682507-5	IS-05 (Flataskóli)

## Analytical methods

### ***Analyses for content of metals by ICP/MS***

Part samples of the commodities were by means of microwave-induced heating prepared with a mixture of HNO<sub>3</sub> (from sub boiling quality) and hydrogen peroxide. The resulting solutions were diluted with Milli-Q water and analysed by ICP/MS.

Analyses in duplicate were carried out for all samples and references, and the calibration will be verified with traceable independent control samples.

Detection limit: 0.05 mg/kg

Estimated uncertainty: 20% RSD

### ***Analyses for content of Phenols, Cresols and Xylenols by GC/MS***

The sub samples of the materials were extracted with hexane/ acetone (80:20) with deuterated internal standard phenol. Analyses of the extracts were performed by capillary gas chromatography combined with mass spectrometry (GC/MS).

The identification of the substances was performed by using the NIST-database.

Detection limit: 5 – 20 mg/kg

Estimated uncertainty: 20% RSD

### ***Analyses for content of 7 congeners of Polychlorinated Biphenyls (PCB) by GC/MS***

The sub samples of the materials were extracted with dichloromethane with deuterated internal standard of Hexachlorobenzene. Analyses for the 7 congeners of PCB were performed by capillary gas chromatography combined with mass spectrometry (GC/MS).

Detection limit: 0.01-0.08 mg/kg

Estimated uncertainty: 35% RSD

### ***Analyses for Phthalates***

The subsamples from the surfaces were extracted with hexane/acetone (80:20) spiked with deuterated internal standards of DBP-d<sub>4</sub> and DEHP-d<sub>4</sub>. Analyses of the extracts for the 6 regulated phthalates according to REACH were performed by capillary gas chromatography combined with mass spectrometry (GC/MS).

The quantifications of the phthalates were performed using standards of the single phthalates and the identifications of the phthalates were performed by using the NIST-database.

Reference method: DS/ISO 16181

Detection limit: 5 – 20 mg/kg

Estimated uncertainty: 20% RSD

### ***Analyses for Polyaromatic hydrocarbons (PAH)***

The subsamples from the surfaces were extracted with hexane/acetone (80:20) spiked with deuterated internal standards. Analyses of the extracts were performed by capillary gas chromatography combined with mass spectrometry (GC/MS).

The quantifications of PAH were performed using standards of the single PAH and the identifications of the PAH were performed by using the NIST-database.

Detection limit: 5 mg/kg

Estimated uncertainty: 35% RSD

## Results

### Quantitative test results of the content analyses by ICP/MS in mg/kg

Unit: mg/kg	Sample				
Metal/element	682507-1	682507-2	682507-3	682507-4	682507-5
Chrome, Cr	10*	1.0	1.2	1.9*	10
Manganese, Mn	43	3.0	3.7	16	81
Nickel, Ni	2.0	1.7*	3.0*	3.6*	3.6*
Copper, Cu	3.0	17*	43	31	50
Zinc, Zn	6500	15000*	14000	14000	14000*
Cadmium, Cd	0.1*	4.4*	0.7	1.1*	1.8
Tin, Sn	0.8*	14*	1.4*	0.63	2.3*
Mercury, Hg	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Lead, Pb	9.3	15	18	20	16
Aluminium, Al	16000*	270*	670*	1000*	1300*

< Means less than the limit of detection.

\* Means that the results of the double determination vary more than the analytical uncertainty.

**Quantitative test results of the content analyses of Phenols, Cresols and Xylenols in mg/kg**

Unit: mg/kg	Sample				
Substance	682507-1	682507-2	682507-3	682507-4	682507-5
Phenol	<5	<5	<5	<5	<5
Cresols	<10	<10	<10	<10	<10
Xylenols	<10	<10	<10	<10	<10

< Means less than the limit of detection.

**Quantitative test results of the content analyses of 7 congeners of PCB in mg/kg**

Unit: mg/kg	Sample				
Substance	682507-1	682507-2	682507-3	682507-4	682507-5
PCB 28	<0.05	<0.05	<0.05	<0.05	<0.05
PCB 52	<0.08	<0.08	<0.08	<0.08	<0.08
PCB 101	<0.05	<0.05	<0.05	<0.05	<0.05
PCB 118	<0.02	<0.02	<0.02	<0.02	<0.02
PCB 138	<0.02	<0.02	<0.02	<0.02	<0.02
PCB 153	<0.02	<0.02	<0.02	<0.02	<0.02
PCB 180	<0.01	<0.01	<0.01	<0.01	<0.01

**Quantitative test results of the content analyses of PAH in mg/kg**

Unit: mg/kg		Sample				
Component	CAS no.	682507-1	682507-2	682507-3	682507-4	682507-5
Naphthalene	91-20-3	<5	<5	<5	<5	<5
Acenaphthylene	208-96-8	<5	<5	<5	<5	<5
Acenaphthene	83-32-9	<5	<5	<5	<5	<5
Fluorene	86-73-7	<5	<5	<5	<5	<5
Phenanthrene	85-01-8	<5	<5	<5	<5	<5
Anthracene	120-12-7	<5	<5	<5	<5	<5
Fluoranthene	206-44-0	<5	<5	<5	<5	<5
Pyrene	129-00-0	<5	<5	<5	<5	<5
Benzo(a)anthracene	56-55-3	<5	<5	<5	<5	<5
Chrysene	218-01-9	<5	<5	<5	<5	<5
Benzo(b,j,k)fluoranthenes 3 Substances	205-99-2 205-83-2 207-08-9	<5	<5	<5	<5	<5
Benzo(a)pyrene	50-32-8	<5	<5	<5	<5	<5
Benzo(e)pyrene	192-97-2	<5	<5	<5	<5	<5
Indeno(1,2,3-cd)pyrene	193-39-5	<5	<5	<5	<5	<5
Dibenzo(a,h)anthracene	53-70-3	<5	<5	<5	<5	<5
Benzo(ghi)perylene	191-24-2	<5	<5	<5	<5	<5

<: Means less than the limit of detection.

The results are the means of the double determinations of each sample.

### Quantitative test results of the content analyses of Phthalates

Unit: mg/kg		Sample				
Component	CAS no.	682507-1	682507-2	682507-3	682507-4	682507-5
Dibutyl phthalate (DBP)	84-74-2	<5	<5	<5	<5	<5
Butylbenzyl phthalate (BBP)	85-68-7	<5	<5	<5	<5	<5
Di(2-ethylhexyl)phthalate (DEHP)	117-81-7	<5	<5	<5	<5	<5
Di-n-octyl phthalate (DNOP)	117-84-0	<5	<5	<5	<5	<5
Diisononyl phthalate isomers (DINP)	68515-48-0 28553-12-0	<20	<20	<20	<20	<20
Diisodecyl phthalate isomers (DIDP)	68515-49-1 26761-40-0	<20	<20	<20	<20	<20

<: Means less than the limit of detection.

The results are the means of the double determinations of each sample.