- Report -

30% MCCP containing pulverised PU foam

Leaching Study, Limit Test

Modified to GHS Rev.5, Annex 10 (2013)
and
OECD No. 29 (2001)

Sponsor

FEICA AISBL
Avenue E. van Nieuwenhuyse 4
1160 Brussels
Belgium

Author

[redacted]

Test Facility

Noack Laboratorien GmbH
Käthe-Paulus-Str. 1
31157 Sarstedt
Germany

Study ID

acc. to GLP

150623HW / CLW16893

Study completed on

29. NOV. 2016

Statement of GLP Compliance

Title	30% MCCP containing pulverised PU foam Leaching Study, Limit Test
Guidelines	Modified to GHS Rev.5, Annex 10 (2013) and OECD No. 29 (2001)
Test Item	30% MCCP containing pulverised PU foam (Batch number 2)
Test Facility	Noack Laboratorien GmbH Käthe-Paulus-Str.1, 31157 Sarstedt, Germany Phone: +49 5066 7067 0, Fax: +49 5066 7067 89 E-mail: info@noack-lab.de

We declare that this study was conducted and reported in compliance with present OECD, EC and German principles of Good Laboratory Practice.

29.11.2016
(Date)

[redacted]

30.11.16
(Date)

[redacted]

Statement of the Quality Assurance Unit

Title 30% MCCP containing pulverised PU foam
Leaching Study, Limit Test

Guidelines Modified to GHS Rev.5, Annex 10 (2013) and OECD No. 29 (2001)

Test Item 30% MCCP containing pulverised PU foam (Batch number 2)

The study was verified and reported to the study director and test facility management as follows:

Inspected study phase		Inspection date	Date of report
Study plan		2016-06-13 2016-08-11	2016-06-13 2016-08-11
Experimental phase	Sampling for analysis	2016-09-13	2016-09-13
	Analysis		
Report		2016-09-28 2016-10-06 2016-11-08 2016-11-29	2016-09-28 2016-10-06 2016-11-08 2016-11-29

The reported results accurately and completely reflect the raw data of the study. Also methods, procedures, and observations are accurately and completely described in the report.

The accordance of the study with its study plan and the principles of Good Laboratory Practice is guaranteed.

23.11.16
(Date)

[redacted]

Personnel Involved

Study Director: [redacted]

Technical Staff: [redacted]

Quality Assurance Unit: [redacted]

[redacted]

[redacted]

Test Facility Management: [redacted]

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List of Abbreviations

CAS	Chemical Abstract Service
cm	Centimeter
GC-MS	Gas chromatography with mass selective detector
GHS	Globally Harmonized System of Classification and Labelling of Chemicals
GLP	Good Laboratory Practice
L	Liter
mg	Milligram
OECD	Organization for Economic Co-operation and Development
QAU	Quality Assurance Unit

1 Summary

The leaching of C14-C17 chloroalkanes out of the test item 30% MCCP containing pulverised PU foam in aqueous media modified to Globally Harmonized System of Classification and Labelling of Chemicals (GHS), Rev. 5, Annex 10 (2013) and OECD No. 29 (2001) was performed from 2016-08-16 to 2016-09-14 at Noack Laboratorien GmbH.
30% MCCP containing pulverised PU foam was prepared by the sponsor as cured and freeze-grinded particles with a size of < 2 mm (99.9%, for details please refer to part 6.1) to increase the surface of the test item. The test item was determined as limit loading of 1 mg/L in ISO Test water (according to OECD 202 Annex 3) for a test period of 28 days. Analytical evaluation of the substances of interest C14-C17 chloroalkanes was performed in triplicate at the end of the exposure phase (28 days) *via* GC-MS/MS by measuring the total concentration of chlorinated alkanes.

The determined concentrations in three replicates and the control were between 0.730 µg/L and < LOQ (limit of quantification of 0.4 µg/L). The results are summarized in Table 1; for details of the analytical method, please refer to part 4.

Table 1: **Summary of the Measured Concentrations of C14-C17 chloroalkanes**

Sampling Date	C14-C17 Chloroalkanes			
	Control	Limit Loading 1 mg/L		
		Replicate 1	Replicate 2	Replicate 3
	Meas. conc. [µg/L]	Meas. conc. [µg/L]	Meas. conc. [µg/L]	Meas. conc. [µg/L]
2016-09-13 (28 d)	< LOQ	0.725[1)]	0.730[2)]	< LOQ

LOQ	Limit of quantification (0.4 µg C14-C17 chloroalkane /L)
Meas. conc.	Measured concentration of C14-C17 chloroalkane
1)	Reanalysed on 2016-09-14, mean value of two samples
2)	Reanalysed on 2016-09-14, mean value of three samples

Measured concentrations of C14-C17 chloroalkanes were below the NOEC of 10 µg/L (lowest NOEC for C14-C17 chloroalkanes for aquatic invertebrates and the corresponding PNEC of 1 µg/L (freshwater) as reported on REACH dissemination Webpage, Reg no.: 01-2119519269-33-0000, status November 2016.

2 Test Item

2.1 Test Item Properties

TEST ITEM	30% MCCP containing pulverised PU foam
Batch number	2
Substance of interest / content	Product contains C14-C17 chloroalkanes (30.4%); chlorine content 45.31%
Preparation of the test item	The preparation of the powder was carried out by the sponsor as follows: A mixture of the raw materials, including C14-C17 chloroalkanes, was placed in a pressure vessel and a propellant mixture of iso-butane and dimetylether was added. The pressure vessel was homogenized by shaking and was kept at room temperature for two days (formation of pre-polymer). Several samples of the foam (beads) were taken (approx. 1 cm Ø) and kept at -18 °C for 24 h until complete hardening. The cured foam was pulverized by grinding.
CAS-No.	85535-85-9
Physical state at room temperature	Solid, powder
Expiry date	2020-11-13
Stability under test conditions	Assumed to be stable
Recommended storage	< 23 °C, protected from light, store dark

The test item and the information concerning it were provided by the sponsor.

2.2 Test Facility Actions

Receipt	2015-11-13
Identification parameters	Name, physical state and consistency
Retention sample	At least 1 g has been retained on 2015-12-01 and stored at 6 ± 2 °C.
Storage conditions	At 6 ± 2 °C, protected from light, in a tightly closed container.

3 Method

TEST GUIDELINES	The study was performed modified to: Globally Harmonized System of Classification and Labelling of Chemicals (GHS), Rev. 5, Annex 10 (2013) "Guidance on transformation/dissolution of metals and metal compounds in aqueous media" and OECD Number 29 (2001) "Guidance document on transformation/dissolution of metal compounds in aqueous media" The study was performed in compliance with GLP. For the respective guidelines please refer to the part 9 'Literature'.
TYPE AND PURPOSE OF THE STUDY	Review of labelling of the product according to Regulation (EC) No. 1272/2008 (CLP). The leaching of the substance of interest into aqueous media in 28 days was measured at concentrations below the NOEC.
TEST SYSTEM	ISO Test water, according to OECD 202, Annex 3 (see test medium)

3.1 Experimental Procedure

Test Item	30% MCCP containing pulverised PU foam
Limit loading	A limit loading of 1 mg 30% MCCP containing pulverised PU foam/L was tested.
Partical size	The determination of the particle size of fractions of 30% MCCP containing pulverised PU foam was part of a non-GLP measurement using analytical sieves with the mesh width 1 mm and 2 mm. For results see section 6.1.
Test solution	The test solution was freshly prepared with test medium prior to application. For preparation see 'application'.

Control	Test medium without test item
Duration	28 days
Replicates	Three replicates of the limit loading per sampling day and one control replicate
Test vessels / volume	Glass bottles / 1000 mL
Test medium	

Table 2: **ISO Test Water, according to OECD 202, Annex 3 and OECD Number 29 (2001)**

Component	Concentration [mg/L]
KCl	5.76
$NaHCO_3$	64.8
$CaCl_2 \cdot 2\ H_2O$	294
$MgSO_4 \cdot 7\ H_2O$	123

Temperature	20 ± 2 °C
pH value	approx. 7.8; see part 6.4
Dissolved oxygen concentration	≥ 5.95 mg/L
Light conditions	Low light conditions
Application	The test vessels were rinsed successively with demineralized water, acetone and n- hexane. After drying, the test medium was filled into the test vessels and the pH value as well as the concentration of dissolved oxygen was measured in each vessel after at least 30 min standing. 1 mg of the test item was weighed out for each test vessel containing 1000 ml test medium. These test vessels as well as control replicate were agitated.
Agitation	Shaker (150 rpm)
Sampling	At least three samples were taken from each test vessel of the limit loading on day 7, 14, 21 and 28. Sampling was performed directly from the test vessels while separating the medium from the test item (left standing for at least 1 h). Samples from the control were

	taken on day 28. Samples were stored at 6 ± 2 °C after sampling and until analysis.
TYPE AND FREQUENCY OF MEASUREMENTS	The total concentration of C14-C17 chloroalkanes (MCCP) of the limit loading (three replicates) and the control were analysed *via* GC-MS/MS at the end of the study (day 28). Additional samples were taken at day 7, day 14 and day 21 from additional replicates and stored at 6 ± 2 °C. The concentration of dissolved oxygen and the pH value was measured at the start (day 0) and at the end (day 28) of the study. The temperature was measured throughout the whole study.
Procedure control	The test medium was spiked threefold with Cerechlor S45 at 1 x LOQ level at the end of the study (day 28) and was analysed with the test samples.
Equipment	Analytical balances (SARTORIUS) Oximeter and pH-Meter, HQ 40d multi (HACH) Shaker (EDMUND BÜHLER) Thermohygrograph (LUFFT) Datalogger (TESTO)
Reagents	Demineralized water, generated in house Magnesium sulphate heptahydrate, $MgSO_4 * 7\ H_2O$, ≥ 99%, batch: 156240496 (ROTH) Sodium hydrogen carbonate, $NaHCO_3$, 99.5%, batch: 076233841 (ROTH) Calcium chloride dihydrate, $CaCl_2 * 2\ H_2O$, ≥ 99%, batch: BCBR8134V (SIGMA-ALDRICH) Potassium chloride, KCl, 99.5%, batch: 475237252 (ROTH)

4 Analytical Method

Analytical evaluation of C14-C17 chloroalkanes was carried out *via* GC-MS/MS by measuring the total concentration of chlorinated alkanes. Matrix matched standards were used for external calibration. Two transitions of the same precursor ion (m/z 102) were measured, which is present in mass spectra of all chloroalkanes.
Furthermore, the particle size of 30% MCCP containing pulverised PU foam was determined by the dry sieving technique using analytical sieves with the mesh width 1 mm and 2 mm (non-GLP).

4.1 Analytical System

Equipment	Gas chromatograph	: 7890B, AGILENT
	Autosampler	: CHRONECT Robotic RTC PAL, liquid, AXEL SEMRAU
	Injector	: Optic-4s, GLSCIENCES
	Detector	: 7010 Quadrupol-MS/MS, AGILENT
	Software	: MassHunter B 07, AGILENT Chronos 4.5.1, AXEL SEMRAU Evolution Workstation 4.6.3, GLSCIENCES
	Inlet Liner	: Fritted Liner with Chromosorb for LVI, GLSCIENCES
	Column	: HP5-MS, 30 m, 0.25 mm ID, 0.25 µm film thickness, batch: USE723126H, AGILENT
Injection	Large Volume Injection, 100 µL	
Injector temperature	55°C, after 82 sec vent time ramping to 300°C with 15°C/sec	

Table 3: **GC Temperature Program**

Temperature [°C]	Heating rate [°C/min]	Hold time [min]
50	0	6
325	40	6

Carrier gas Helium	2.0 mL/min
Run time	18.875 min
Retention time	Approx. 10-16 min

Conditions of detection	Source Temperature	230 °C
	Ionisation mode	Electron ionization (EI), positive
	Scan mode	Centroid
	Scan method	MS/MS
Quantification/ Reference ions	m/z 102->67 (quantification), 102->65 (reference)	
Additional equipment	Positive-displacement pipette, Microman (GILSON MEDICAL)	
Additional reagents	n-Hexane, ≥ 95%, VWR Sodium sulphate, ≥ 99%, SIGMA-ALDRICH Acetone, ≥ 99.7%, VWR	
Analytical Standard	C14-C17 chloroalkanes, Cereclor S45, batch: 788432, chlorine content: 45.31%, INEOS ChlorVinyls, provided by the sponsor	

4.2 Sample Preparation

Preparation of standards	Stock solutions of 500 mg/L Cereclor S45 were prepared in acetone and were diluted to 10 mg/L with n-hexane and further to 7 concentrations. From these solutions, 10 µL was added to 990 µL of matrix-matched n-hexane. The matrix-matched n-hexane was prepared by extraction of the test medium (control) as described under 'preparation of samples'. The resulting solutions were used for calibration. For calibration range refer to part 4.3
Preparation of samples for method validation and quality control	Stock solutions of 500 mg/L Cereclor S45 in acetone were diluted to concentrations of 400 µg/L (for 1 x LOQ and quality control) and 800 µg/L (for 2 x LOQ) in acetone. 100 µL of these solutions were pipetted into 100 mL test medium. The aqueous solution was extracted for 2 min with 5 mL n-hexane. The organic phase is separated and dried with Na_2SO_4 and measured without further dilution.
Preparation of samples	Samples were taken as described above for 'sampling'. The aqueous solution was extracted for 2 min with 5 mL n-hexane. The organic phase is separated and dried with Na_2SO_4 and measured without further dilution.

4.3 Validation of Analytical Method

The analytical method was validated regarding the aspects shown in OECD No. 29 and according to SANCO 3029/99 rev.4 (2000) using the following criteria:

Table 4: **Parameter, Acceptance Criteria and Results of the Method Validation**

Parameter	Acceptance criteria	Result	
Linearity	At least 5 standard concentrations, $r^2 \geq 0.992$	2 to 30 µg C14-C17 chloroalkanes/L (n = 7), $r^2 \geq 0.992$	✓
Lowest calibration level	S/N ≥ 9 for quantifier ion trace	2 µg C14-C17 chloroalkanes/L, S/N: 31	✓
Limit of Detection (LOD)	S/N ≥ 3 for quantifier ion trace	Not determined based on S/N for LOQ ≥ 30	✓
Limit of Quantification (LOQ)	0.4 µg C14-C17 chloroalkanes (Cereclor S45)/L	0.4 µg C14-C17 chloroalkanes (Cereclor S45)/L	✓
Accuracy (Fortified samples)	Mean recovery rate of 70-110% (ideally 80-100%) per fortification level (2 levels)	1 x LOQ: 98 % (n = 5) 2 x LOQ: 104 % (n = 5)	✓
Precision	Relative standard deviation ≤ 20% per fortification level	1 x LOQ: 15 % 2 x LOQ: 11 %	✓
Specificity (GC-MS/MS)	Measurement of two transitions of the same precursor ion - one quantifier (used for evaluation) and one qualifier (for confirmation of the analyte identity)	m/z 102->67 (quantifier) m/z 102->65 (qualifier)	✓
	Blank values < 30 % of the LOQ	Blank values < 30 % of LOQ	

✓ Criterion fulfilled

The results of accuracy and precision are presented in Table 5 and demonstrate the validity of the analytical method. Mean recoveries of 98% at 1 x LOQ and 104% at 2 x LOQ were obtained. The corresponding coefficients of variation were ≤ 15%, indicating a sufficient precision of the analytical method. Since chlorinated alkanes are ubiquitous, the analytical background had to be corrected by using matrix-matched calibration standards.

Table 5: **Fortified Samples**

Fortified concentrations: 0.4 µg/L C14-C17 chloroalkanes (1 x LOQ), 0.8 µg/L C14-C17 chloroalkanes (2 x LOQ)

Replicate	C14-C17 chloroalkanes			
	1 x LOQ		2 x LOQ	
	Meas. conc. [µg/L]	%	Meas.conc. [µg/L]	%
1	0.412	110	0.761	93
2	0.485	118	0.935	114
3	0.323	79	0.714	87
4	0.384	94	0.830	101
5	0.400	98	1.04	127
Mean	0.40	98	0.9	104
SD	0.06		0.1	
CV [%]	15		11	

Meas. conc. = Measured concentration
% = Percent of fortified concentration
SD = Standard deviation
CV = Coefficient of variation

5 GLP Issues

Dates	Study initiation	2016-08-11
	Experimental starting	2016-08-16
	Experimental completion	2016-09-14
	Study completion	Please refer to page 1

Chronological test description

- Method Validation
- Application (experimental starting)
- Measurement of the pH value and the concentration of dissolved oxygen (day 0 and day 28)
- Determination of the concentrations of the substance of interest at the end of the study (day 28)
- Evaluation of data

Deviations from the test guideline

The study was performed using test medium without regarding the pH value, since no effect of pH on solubility of the substance of interest is expected.

Deviations from the study plan

none

Archiving

The following will be retained in the archive of the test facility for the period as specified in the operative national GLP regulations:

- all raw data
- study plan
- final report
- all records performed by the quality assurance programme including master schedules
- sample of test item

6 Results

6.1 Particle Size (non-GLP)

The determination of the particle size of fractions of 30% MCCP containing pulverised PU foam was part of a non-GLP measurement using analytical sieves with the mesh width 1 mm and 2 mm. The empty sieves and a receiver pan were weighed out and the weight was noted. 50 g of the test item were weighed out in the 2 mm sieve, which was covered with the receiver pan. The stack was mounted on the sieving machine and sieving was performed for 15 min with an amplitude of 1.5 mm. Afterwards, the sieve was weighed out and sieving was repeated for 5 min intervals until the weighing of two sequent sieving intervals differ less than 0.1 g. The procedure was repeated as described above covering the 1 mm sieve. The results are shown in Table 6.

Table 6: **Particle Size Distribution of 30% MCCP containing pulverised PU foam (non-GLP)**

Particle size fraction [mm]	Mass fraction [%]
≥ 2	0.08
< 2 - ≥ 1	5.07
< 1	94.81

6.2 Temperature

The room temperature was monitored with a datalogger or thermohygrograph. The temperature was kept at 20 ± 2 °C during the 28 days.

6.3 Dissolved Oxygen Concentration

The dissolved oxygen concentration measured at the start (day 0) and at the end (day 28) of the study was > 5.95 mg/L. Results are given in Table 7.

Table 7: **Dissolved Oxygen Concentration in the Test Vessels**

Date	2016-08-16 (0 d)	2016-09-13 (28 d)
30% MCCP containing pulverised PU foam Limit Loading 1 mg/L	Dissolved oxygen concentration [mg/L]	
Control	7.01	8.17
Replicate 1	7.03	7.61
Replicate 2	7.07	7.84
Replicate 3	7.17	8.01

6.4 pH Value

The pH value was measured at the start (day 0) and at the end (day 28) of the study and was between 6.97 and 7.94. Exact values are given in the following table.

Table 8: **pH Values in the Test Vessels**

Date	2016-08-16 (0 d)	2016-09-13 (28 d)
30% MCCP containing pulverised PU foam Limit Loading 1 mg/L	pH value	
Control	7.64	6.92
Replicate 1	7.94	7.11
Replicate 2	7.90	7.00
Replicate 3	7.90	6.97

6.5 Concentration of C14-C17 chloroalkanes

Samples of the limit loading level of 1 mg/L and the control of the test item 30% MCCP containing pulverised PU foam were analysed at the end of the exposure phase (day 28). Analytical evaluation of the substances of interest C14-C17 chloroalkanes was performed from three replicates and the control *via* GC-MS/MS by measuring the total concentration of chlorinated alkanes. The determined concentrations were between 0.730 µg/L and < LOQ (limit of quantification of 0.4 µg/L) in the replicates and < LOQ in the control. Measurement of three replicates of 1 x LOQ level as procedure control indicated validity of the analytical method. The results are shown in Table 9 and Table 10. Since the concentrations after 28d were below the lowest NOEC data reported for C14-C17 chloroalkanes, no further samples (7 d, 14 d or 21 d) were determined.

Table 9: **Determined Concentrations of C14-C17 Chloroalkanes**

Sampling Date	C14-C17 Chloroalkanes			
	Control	Limit loading 1 mg/L		
		Replicate 1	Replicate 2	Replicate 3
	Meas. conc. [µg /L]	Meas. conc. [µg /L]	Meas. conc. [µg /L]	Meas. conc. [µg /L]
2016-09-13 (28 d)	< LOQ	0.725[1)]	0.730[2)]	< LOQ

LOQ = Limit of quantification (0.4 µg C14-C17 chloroalkane /L)
Meas. conc. = Measured concentration of C14-C17 chloroalkane
1) = Reanalysed on 2016-09-14, mean value of two samples
2) = Reanalysed on 2016-09-14, mean value of three samples

Table 10: **Measured Concentrations of Procedure Controls**

Nominal concentration: 0.4 µg/L C14-C17 chloroalkanes

Replicate	C14-C17 chloroalkanes	
	Analyses on 2016-09-13 (day 28)	
	Meas.conc. [µg/L]	%
1	0.398	99
2	0.260	65
3	0.437	109
Mean	0.365	**91**

Meas. conc. = Measured concentration of C14-C17 chloroalkanes
% = Percent of the nominal concentration

7 Validity Criteria

The study was conducted modified to Globally Harmonized System of Classification and Labelling of Chemicals (GHS), Rev. 5, Annex 10 (2013) and OECD No. 29 (2001). The concentration of C14-C17 chloroalkanes was below the limit of quantification (LOQ) of 0.4 µg/L in one replicate. Concentrations above the LOQ were measured in two replicates of the limit loading. The measured concentrations were 0.725 µg/L and 0.730 µg/L and were therefore comparable and plausible. For calculation of a variation, the LOQ was taken into account for the third replicate. The variation (relative standard deviation) was 31%. However, since two replicates showed comparable concentrations and the variation has to be calculated for at least three replicates, the study is estimated as valid.

8 Conclusions

30% MCCP containing pulverised PU foam was prepared by the sponsor as cured and freeze-grinded particles with a size of < 2 mm (99.9%, for details please refer to part 6.1) to increase the surface of the test item. The test item was determined as limit loading of 1 mg/L in ISO Test water, according to OECD 202, Annex 3 for a test period of 28 days. Analytical evaluation of the substances of interest C14-C17 chloroalkanes was performed from three replicates and the control *via* GC-MS/MS at the end of the exposure phase.
Measured concentrations of C14-C17 chloroalkanes were below the NOEC of 10 µg/L (lowest NOEC for C14-C17 chloroalkanes for aquatic invertebrates and the corresponding PNEC of 1 µg/L (freshwater) as reported on REACH dissemination Webpage, Reg no.: 01-2119519269-33-0000, status November 2016.

9 Literature

- OECD Principles on Good Laboratory Practice (as revised in 1997), ENV/MC/Chem(98)17, Environment Directorate, OECD, Paris, 1999
- Directive 2004/10/EC, The OECD Principles of Good Laboratory Practice (GLP)
- Principles of Good Laboratory Practice – German Chemical Law (ChemG), Annex 1
- SANCO/3029/99 rev.4, Residues: Guidance for generating and reporting methods of analysis in support of pre-registration data requirements for Annex II (part A, Section 4) and Annex III (part A, Section 5) of Directive 91/414 (11/07/00)
- Globally Harmonized System of Classification and Labelling of Chemicals (GHS), Rev. 5, Annex 10, 2013 "Guidance on transformation/dissolution of metals and metal compounds in aqueous media"
- Regulation on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006 (CLP)
- OECD-Guidance Document No. 29 on Transformation/Dissolution of Metal Compounds in Aqueous Media (2001)
- ECHA Webpage for C14-C17 chloroalkanes (Reg No. 01-2119519269-33-0000). 2016. https://echa.europa.eu/registration-dossier/-/registered-dossier/15252/6/2/5

10 Representative Calibration Curve

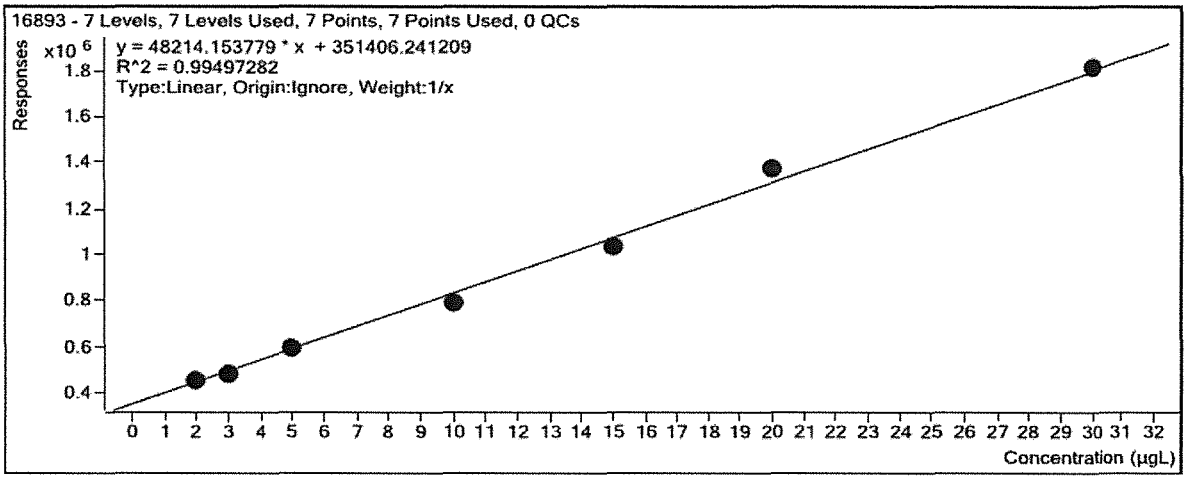


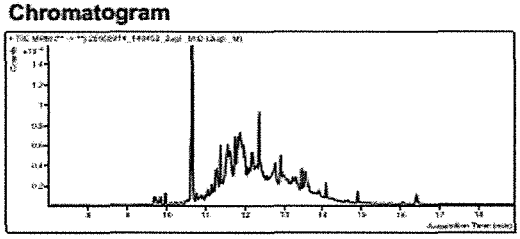
Figure 1: **Calibration of the analytical Standard Cereclor S45**
(dated 2016-09-14)

11 Representative Chromatograms

Analysis Info

Data File	20160914_140452_2ugl _M.D
Acq Time	2016-09-14 14:10
Position	Tray Holder 2:Slot2:20
Sample Name	2ugl _M
Inj Vol	100
Acq Method File	O_16893_MRM_ei_Hexan_lvi_short_50_160728
Sample Type	Calibration

Sample Chromatogram



Quantitation Results

Compound	RT	Response	Conc	Accuracy
16893	10,641	458711	2,2256	111,28

Compound Graphics

Target Compound 16893

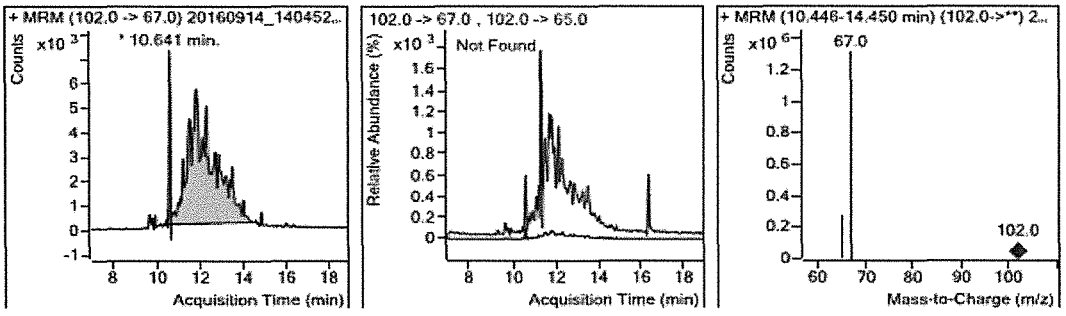
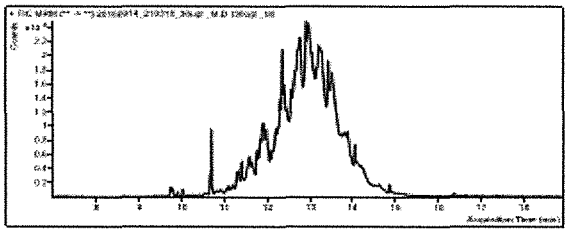


Figure 2: **Chromatograms of the Lowest Analytical Standard**
0.2 µg/L (dated 2016-09-14)

Analysis Info

Data File	20160914_210318_30ugl _M.D
Acq Time	2016-09-14 21:08
Position	Tray Holder 2:Slot2:26
Sample Name	30ugl _M
Inj Vol	100
Acq Method File	O_16893_MRM_ei_Hexan_lvi_short_50_160728
Sample Type	Calibration

Sample
Chromatogram



Quantitation Results

Compound	RT	Response	Conc	Accuracy
16893	13,792	1813096	30,3166	101,06

Compound Graphics
Target Compound 16893

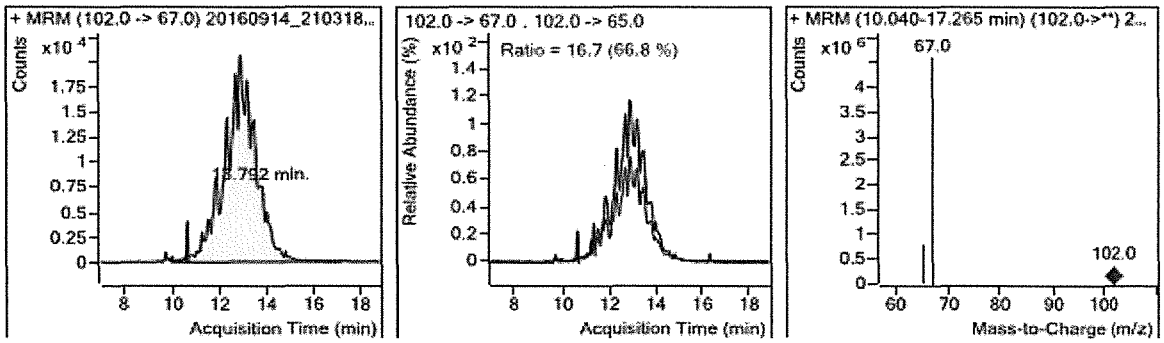
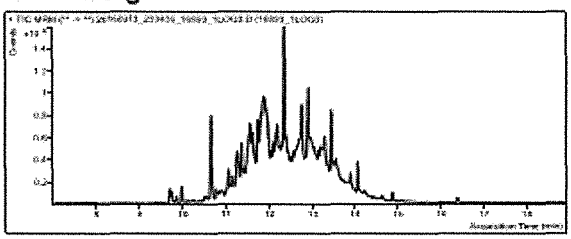


Figure 3: **Chromatograms of the Highest Analytical Standard**
30 µg/L (dated 2016-09-14)

Analysis Info

Data File	20160913_233436_16893_1LOQ3.D
Acq Time	2016-09-13 23:40
Position	Tray Holder 2:Slot2:33
Sample Name	16893_1LOQ3
Inj Vol	100
Acq Method File	O_16893_MRM_ei_Hexan_lvi_short_50_160728
Sample Type	Sample

Sample
Chromatogram



Quantitation Results

Compound	RT	Response	Conc	Accuracy
16893	12,361	720985	8,6968	

Compound Graphics
Target Compound 16893

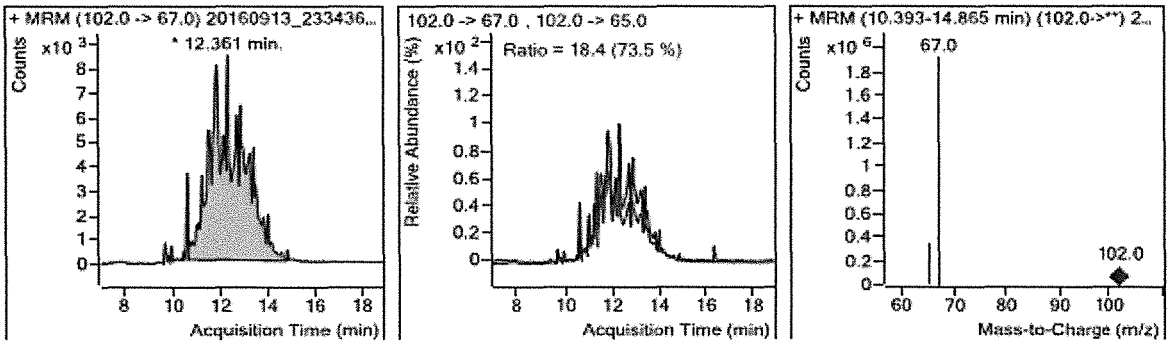
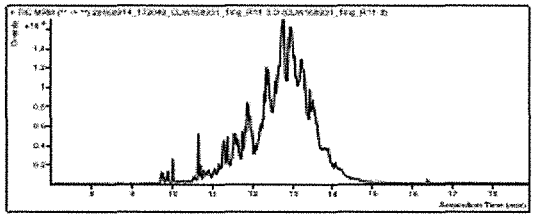


Figure 4: **Chromatogram of a Procedure Control**
0.4 µg/L (dated 2016-09-13)

Analysis Info

Data File	20160914_172049_CLW168931_1mg_R11-3.D
Acq Time	2016-09-14 17:26
Position	Tray Holder 2:Slot2:37
Sample Name	CLW168931_1mg_R11-3
Inj Vol	100
Acq Method File	O_16893_MRM_ei_Hexan_lvi_short_50_160728
Sample Type	Sample

Sample
Chromatogram



Quantitation Results

Compound	RT	Response	Conc	Accuracy
16893	12,749	1122782	15,9990	

Compound Graphics
Target Compound 16893

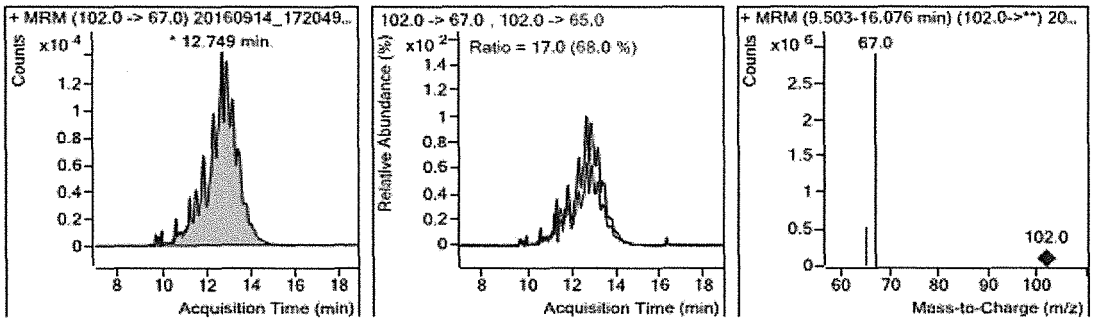
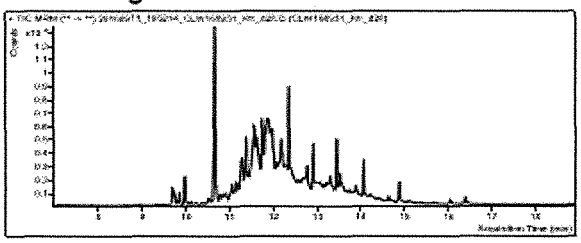


Figure 5: **Chromatogram of a Test Item Replicate at Day 28**
Replicate 2 (dated 2016-09-14)

Analysis Info

Data File	20160913_195214_CLW168931_Ktr_d28.D
Acq Time	2016-09-13 19:57
Position	Tray Holder 2:Slot2:27
Sample Name	CLW168931_Ktr_d28
Inj Vol	100
Acq Method File	O_16893_MRM_ei_Hexan_lvi_short_50_160728
Sample Type	Sample

Sample
Chromatogram



Quantitation Results

Compound	RT	Response	Conc	Accuracy
16893	10,654	409155	2,3835	

Compound Graphics
Target Compound 16893

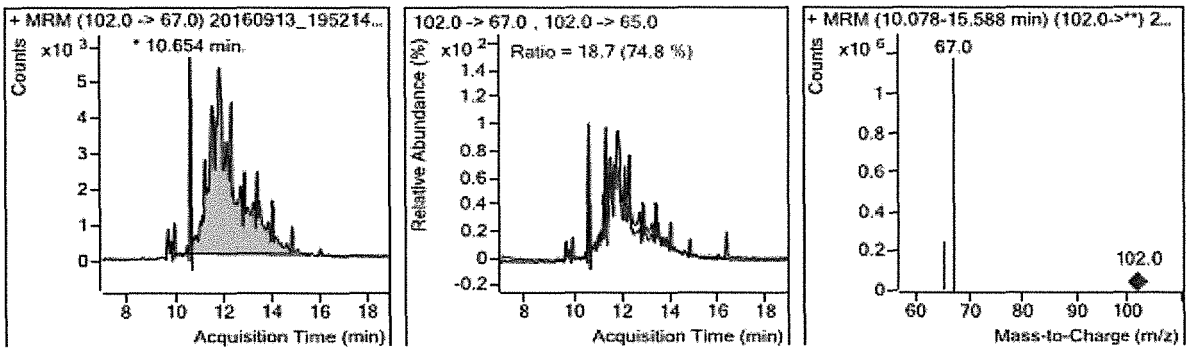


Figure 6: **Chromatogram of the Control at Day 28**
(dated 2016-09-13)

12 Certificates of Analysis

12.1 Certificate of Analysis of the Test Item

Henkel Excellence is our Passion

EINGEGANGEN
13. Nov. 2015
he

Leaching Test 30 % MCCP – Herstellbeschreibung der Probe

1. Herstellung des Prepolymers

   Zu einem Gemisch aus

- 40 g	Polyethertriol (MW 1000)	(CAS 25791-96-2)
- 70 g	Polypropylenglykol (MW 400)	(CAS 25322-69-4)
- 204 g	Chlorparaffin C14-C17 (45 % Chlor)	(CAS 85535-85-9)
- 7 g	silikonbasierter Schaumstabilisator	
- 2 g	2,2-Dimorpholinodiethylether	(CAS 6425-39-4)

   werden in einem Druckbehälter („Aerosoldose")

- 348 g	technisches MDI	(CAS 9016-87-9)

   gegeben. Die Dose wird mit einem Ventil verschlossen und mit den Treibmitteln

- 35 g	Isobutan
- 70 g	Dimethylether

   beaufschlagt.

   Die Dose wird geschüttelt zur Homogenisierung des Gemischs und zwei Tage lang bei Raumtemperatur aufbewahrt zur Abreaktion der Komponenten (Prepolymerbildung).

   Daraus resultiert eine Chlorparaffin (MCCP) Konzentration von **30,4 %** im Prepolymer.

2. Herstellung der Probe

   Durch das Ventil werden mehrere Schaumproben in Raupenform entnommen (ca. 1 cm Ø) und bei -18 °C 24 h lang zur Aushärtung gelagert. Anschließend werden die spröden Raupen bei -18 °C manuell in einem Mörser pulverisiert.

12.2 Certificate of Analysis of Cereclor S45 (External Standard)

INEOS ChlorVinyls

INEOS CHLORVINYLS LIMITED
RUNCORN SITE
RUNCORN, CHESHIRE, WA7 4JE
UK

CERECLOR
CERTIFICATE OF ANALYSIS

GRADE CERECLOR :- Cereclor S45

CUSTOMER BRENNTAG LATVIA SIA
FAX No
QUANTITY 23060KGS
SHIPMENT REF. No 81057456
BATCH No 1252053
BATCH No

DATE 03/04/2014
CUSTOMER ORDER No
RECIPIENT NAME

I HEREBY CERTIFY THAT THE MATERIAL SUPPLIED TO YOU AGAINST THE ABOVE ORDER NUMBER CONFORMS TO THE ANALYSIS SHOWN BELOW. (DENSITY AND VISCOSITY @ 25°C (77°F) UNLESS OTHERWISE STATED)

CHARACTERISTICS		TEST METHOD	TEST RESULTS	
			1252053	
COLOUR	HAZEN	ASTM D1209	50	
VISCOSITY	POISE	ASTM D445	1.98	
DENSITY	g/ml	ASTM D1475 (UK)	1.168	
CHLORINE CONTENT	% w/w	CK/CER/12.3 (UK)	45.31	

APPEARANCE CLEAR LIQUID FREE FROM VISIBLE SUSPENDED MATTER

COMMENTS

D.O.M.03/04/14

Validity, 2 years from date of manufacture if stored in accordance with INEOS ChlorVinyls recommendations

EINGEGANGEN
29. April 2014
[redacted]

bezieht sich auf andere Charge von Cereclor S45 (zur gelieferten Charge 788432 ist kein AZ verfügbar) 29.04.14/he

13 GLP-Certificate of Noack Laboratorien GmbH

Gewerbeaufsicht In Niedersachsen

Staatliches Gewerbeaufsichtsamt Hildesheim

Gute Laborpraxis / Good Laboratory Practice

GLP-Bescheinigung / Statement of GLP Compliance

(gemäß / according to § 19 b Abs.1 Chemikaliengesetz)

Eine GLP-Inspektion zur Überwachung der Einhaltung der GLP-Grundsätze gemäß Chemikaliengesetz bzw. Richtlinie 2004/9/EG wurde durchgeführt in:

Assessment of conformity with GLP according to Chemikaliengesetz and Directive 2004/9/EC at:

☒ Prüfeinrichtung / Test facility ☐ Prüfstandort / Test site

Noack Laboratorien GmbH Käthe-Paulus-Str. 1 31157 Sarstedt DEUTSCHLAND	**Noack Laboratorien GmbH** Käthe-Paulus-Str. 1 31157 Sarstedt GERMANY

Prüfungen nach Kategorien / Areas of Expertise (gemäß / according ChemVwV-GLP Nr. 5.3/OECD guidance)

1 - Prüfungen zur Bestimmung der physikalisch-chemischen Eigenschaften und Gehaltsbestimmungen	1 - physical-chemical testing
4 - Ökotoxikologische Prüfungen zu Bestimmung der Auswirkungen auf aquatische und terrestrische Organismen	4 - environmental toxicity studies on aquatic and terrestrial organisms
5 - Prüfungen zum Verhalten im Boden, im Wasser und in der Luft, Prüfungen zur Bioakkumulation und zur Metabolisierung	5 - studies on behaviour in water, soil and air; bioaccumulation
6 - Prüfungen zur Bestimmung von Rückständen	6 - residue studies
7 - Prüfungen zur Bestimmung der Auswirkungen auf Mesokosmen und natürliche Ökosysteme	7 - studies on effects on mesocosms and natural ecosystems
9 - Sonstige Prüfungen: Prüfungen zur Gentoxizität und Mutagenität in vitro	9 - other studies: studies of genotoxicity and mutagenicity in vitro

Ort / Place: **Sarstedt**

Datum der Inspektion / Date of Inspection (Tag.Monat.Jahr / month.day.year): **03. – 05. Juni 2013 / Jun 03rd – Jun 05th, 2013**

Die/Der genannte Prüfeinrichtung/Prüfstandort befindet sich im nationalen GLP-Überwachungsverfahren und wird regelmäßig auf Einhaltung der GLP-Grundsätze überwacht.

Auf der Grundlage des Inspektionsberichtes wird hiermit bestätigt, dass in dieser Prüfeinrichtung/diesem Prüfstandort die oben genannten Prüfungen unter Einhaltung der GLP-Grundsätze durchgeführt werden können.

The above mentioned test facility/test site is included in the national GLP Compliance Programme and is inspected on a regular basis.

Based on the inspection report it can be confirmed, that this test facility/test site is able to conduct the aforementioned studies in compliance with the Principles of GLP.

Hildesheim, 01.02.2016

Staatliches Gewerbeaufsichtsamt Hildesheim
Im Auftrage

[redacted]