

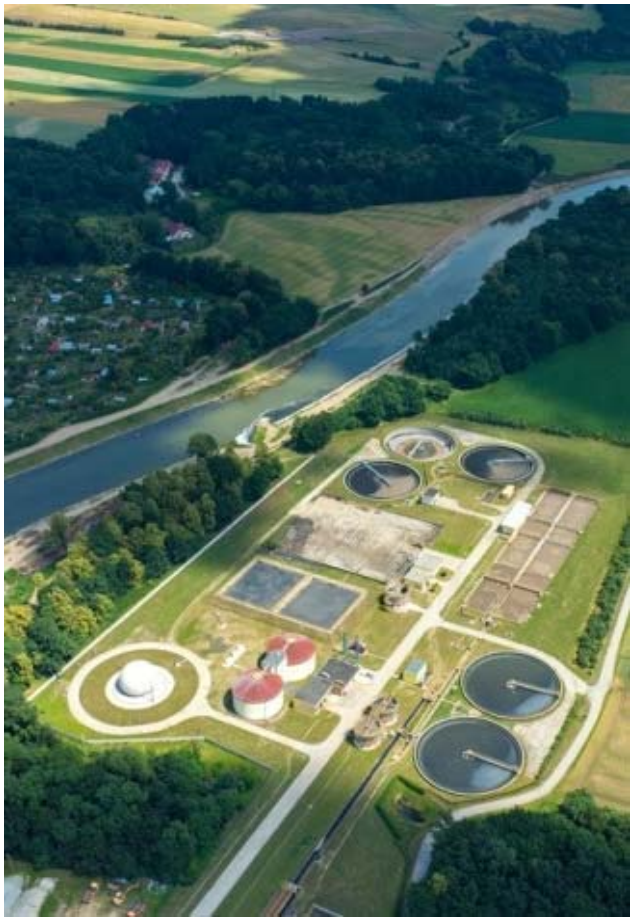

Prepared for the Members of Centre Européen des Silicones (CES)

Periodic Update Report #2 for cVMS (D4 & D5) Restriction Monitoring in EU Program

ERM Study Number: 0383878
R005-0383878
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Date: 2018-06-07

EXECUTIVE SUMMARY

On 11 January 2018, a restriction on the use of octamethylcyclotetrasiloxane (D4) and decamethylcyclopentasiloxane (D5) in wash-off personal care products (PCPs) was published in the Official Journal (EC, 2018). After 31 January 2020, neither D4 nor D5 may be placed on the market in wash-off PCPs in a concentration greater than or equal to 0.1% by weight. The goal of the proposed restriction is to reduce environmental concentrations of D4 and D5 by 78% and 97%, respectively, by significantly controlling the main source of these substances to wastewater and, hence, the aquatic environment – the use of wash-off personal care products containing D4 and D5 (Annex XV Restriction Report, UK Health & Safety Executive, June 2015). The restriction is expected to substantially reduce the concern for aquatic environments receiving discharges from wastewater treatment plants (WWTPs), while permitting other uses that do not pose a concern.

To monitor the effectiveness of the restriction on the use in wash-off PCPs, CES – Silicones Europe (CES), as the project sponsor, and Environmental Resources Management (ERM), as the lead technical consultant, developed a study plan to monitor the concentrations and mass loadings of D4 and D5 in WWTP influent during three, one-year periods: Initial, Transitional, and Post-restriction.

The study plan calls for samples to be collected from six representative WWTPs across the European Union (EU) receiving wastewater from primarily residential sources.

Country	*Site Name and Location*
Germany	Kläranlage Halle Nord (Halle an der Saale, Germany [DE])
Germany	Wolfsburg-Brackstedt (Wolfsburg, DE)
Poland	Stalowa Wola Wastewater Treatment Works (Stalowa Wola, Poland [PL])
Spain	Lleida Wastewater Treatment Works (Lleida, Spain [ES])
Sweden	Norrköping Vatten och Avfall (Norrköping, Sweden [SE])
United Kingdom	Bury Wastewater Treatment Works (Bury, United Kingdom [UK])

Based on an analysis of statistical power, these plants will be sampled eight times per period using a stratified random sampling design that considers potential seasonal, weekly, and daily factors. In addition to D4 and D5, samples will be analyzed for temperature, conductivity, total and volatile suspended solids, and total and dissolved organic matter.

Following the completion of each sampling period (i.e., Initial, Transitional, and Post-restriction), the mass loading of D4 and D5 to these plants will be calculated based on influent flow measured at the time of sampling and reported on a per capita basis based on population data available from the EUROSTAT database. The statistical design allows for a rigorous statistical evaluation of the D4 and D5 data over time to assess the efficacy of the

targeted use restriction (e.g., test of significant difference between the Initial, Transitional and Post-restriction periods).

In addition, following the completion of the Initial, Transitional, and Post-restriction sampling periods, respectively, a correlation model will be developed to account for regional, cultural, and socio-economic factors that influence the use of wash-off PCPs and allow for the results to be extrapolated across the entire EU to estimate the mass loading of D4 and D5 to the environment via wastewater influent. A quantitative comparison to the baseline use of D4 and D5 in wash-off personal care products will be made after each sampling period.

The Study Plan details the methods, materials, and procedures used to collect and analyze influent samples for D4 and D5. The plan considers the experience and expertise developed during a comprehensive exposure assessment conducted in the United States by ERM, on behalf of the Silicone Environmental Health and Safety Council (SEHSC), under an Enforceable Consent Agreement (ECA) with the United States Environmental Protection Agency (USEPA). Additionally, a detailed health and safety plan was prepared to identify all potential hazards (physical, chemical and biological) that may be encountered during sampling and best practices to ensure the safety of the workers involved in collecting and processing the samples. A quality assurance project plan specifies the quality assurance/quality control (QA/QC) samples (e.g., field blanks, field duplicates, field spikes, lab procedural blanks, and calibration standards) and procedures to ensure the data collected are reliable (i.e., consistent, repeatable and credible). Following each sampling event, field documentation will be reviewed for accuracy and completeness and the laboratory reports are reviewed to verify the results are consistent with the method performance criteria and are useable for the purposes of the study. Any deviations and corrective actions are fully documented.

Synlab (Rotterdam, NL), formerly known as ALcontrol, was selected as the analytical laboratory for this program. A laboratory method was developed based on Knoerr et al. (2017) and reviewed by ERM and in-house laboratory personnel from CES member companies. Synlab acquired dedicated equipment, set aside dedicated laboratory space, and received training on how to minimize cross-contamination and conduct the extractions and analyses by member company experts. A laboratory validation study was conducted, including the determination of the method detection limit, demonstration of clean procedural blank samples, and an inter-laboratory calibration study. After the laboratory demonstrated competency in analyzing for D4 and D5, they tested various environmental samples to provide additional training and experience for the laboratory personnel involved in the program. As discussed above, various QA/QC checks are incorporated in the program to ensure acceptable analytical performance, and laboratory audits are conducted at least once per period to verify compliance with all of the necessary procedures and reporting requirements.

A proof-of-concept pilot study was conducted over a five-day period at the Bury, Greater Manchester, UK WWTP. Eight sampling events were conducted, including all combinations of weekday/weekend and time of day (morning, afternoon, evening, and night). Field teams for all six sites were present and alternated between sampling events. In addition, CES member company personnel, familiar with sampling and analysis of D4 and D5, ERM team with experience in the ECA program in the US, and laboratory personnel were present during the pilot study. The pilot study provided valuable training prior to initiating the restriction monitoring program, real-world experience collecting and analyzing samples for D4 and D5, and an opportunity to benefit from lessons learned during the pilot study. The Study Plan was revised based on the lessons learned. Field documentation and laboratory reports were reviewed following completion of the pilot study, and results were compared to method performance criteria and the data were found to be usable for the purposes of this study. The pilot study demonstrated that the Study Plan, including the experimental design and the sampling and analysis procedures, will provide reliable data and accomplish the stated objectives of the restriction monitoring program.

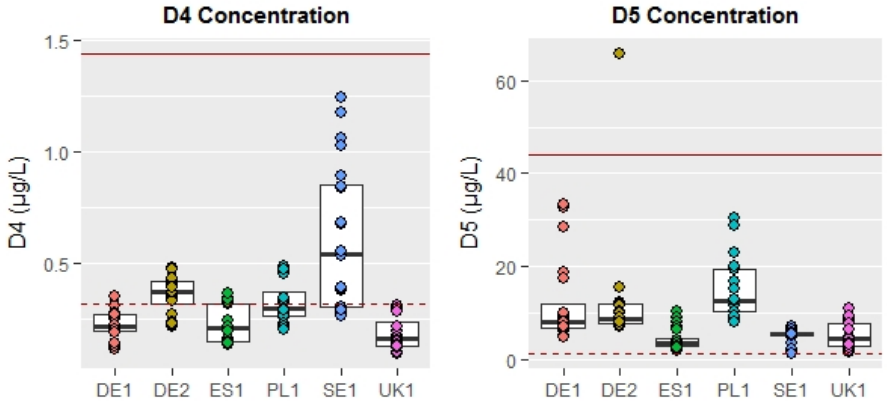
Following the successful completion of the proof-of-concept pilot study, the use restriction monitoring program was initiated in the Fall season of 2017. At the time of this report, five sampling events at the six WWTPs have been completed; however, analytical results from only the first four samplings event are available. Field documentation and laboratory reports were reviewed following completion of the first four sampling events of the Initial Period, and results were compared to method performance criteria and the data were found to be usable for the purposes of this study. The second sampling event for the Spring 2018 season (Sampling Event 6) is scheduled for June, 2018. All of the sampling for the Initial Period will be completed by end of September, 2018. Following the review and analysis of the Initial Period data, the Study Plan may be amended to better address the objectives of the D4 and D5 restriction monitoring program, as appropriate, prior to the start of the Transitional Period, scheduled to start in the Winter season, 2019.

Over the first four sampling events of the Initial Period, a total of 120 influent samples (i.e., 20 primary and unspiked split samples from 6 WWTPs) have been collected and analysed for D4, D5, and matrix characterization parameters (i.e., total organic carbon [TOC], dissolved organic carbon [DOC], total suspended solids [TSS], and volatile suspended solids [VSS]) representing different external factors that can influence the variability of D4 and D5 in WWTP influent, including:

- Season (Spring, Summer, Fall, and Winter);
- Day of week (weekday and weekend); and
- Time of day (morning, afternoon, evening, and night).

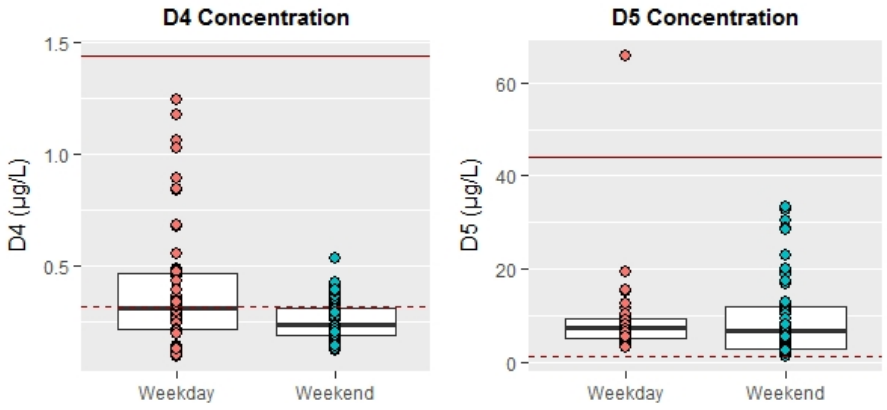
The following plots present the sample results collected over Events 1 through 4 by sampling location and these external factors.

Figure ES1. D4 and D5 concentrations by location – Events 1 - 4



Over the first 4 sampling events of the Initial Period, concentrations of D4 and D5 were typically lower than predicted baseline concentrations at all 6 of the select WWTPs (Figure ES1). Predicted baseline concentrations (D4 = 1.44 µg/L; D5 = 44.1 µg/L) are shown as solid lines and predicted post-restriction concentrations (D4 = 0.32 µg/L; D5 = 1.32 µg/L) are shown as dotted lines on the D4 and D5 concentration plots. Predicted influent concentrations are estimated based on the baseline mass loadings (EA 2105a and EA 2015b) and estimated reduction goals for D4 and D5 (UK Health & Safety Executive 2015), total population of the EU (511,522,671; Eurostat 2017), and the per capita use of water (200 liters per person per day; ECB 2003). The highest D4 concentrations typically occurred at the Norrköping (SE1) WWTP. D5 concentrations measured during Events 1 through 4, were consistently above the predicted post-restriction concentration (Figure ES1). The highest D5 concentrations occurred at the Halle an der Saale (DE1), Wolfsburg (DE2), and Stalowa Wola (PL1) WWTPs.

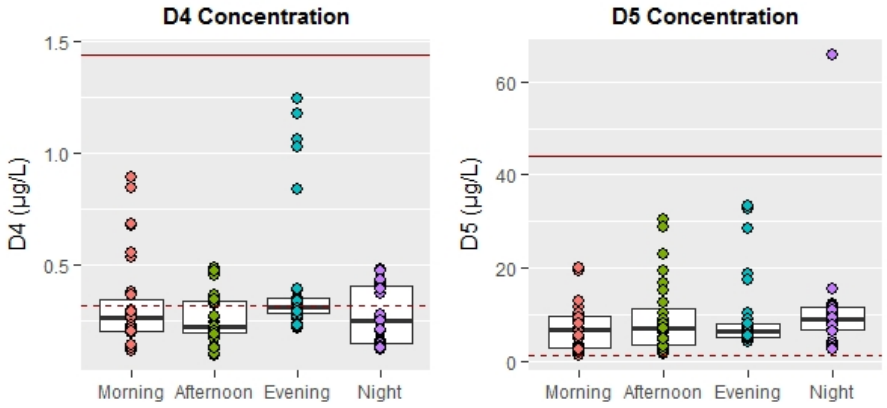
Figure ES2. D4 and D5 concentrations by day of the week – Events 1 - 4



When the data results are grouped and evaluated by the time of week (i.e., collection during weekdays or weekends), notably higher D4 concentrations are associated with samples collected during weekdays. D5 concentrations

measured during Events 1 through 4 were fairly similar between weekday and weekend sampling events (Figure ES2).

Figure ES3. D4 and D5 concentrations by time of day – Events 1 - 4



When the data results are grouped and evaluated by the time of day (i.e., morning, afternoon, evening, or night), concentrations of D4 and D5 are generally similar (Figure ES3). Notably higher individual concentration results were observed during morning and evening (D4) and afternoon, evening, and night (D5) sampling events.

The first four sampling events of the Initial period were collected during the Fall and Winter seasons, a full evaluation of D4 and D5 data on a seasonal basis is not possible until the completion of the Initial Period sampling.

Although it is too early to draw any definitive conclusions, the conclusion that the methods, materials and procedures detailed in the Study Plan will provide reliable data to assess the efficacy of the use restriction is supported by the results from the pilot study and the first four sampling events.

The restriction monitoring program, by sampling WWTP influent and accounting for various internal and external factors that may influence the use of wash-off PCPs, will produce a large and reliable database of D4 and D5 concentrations. These data will be used to support a quantitative assessment of the efficacy of the target use restriction proposed for D4 and D5 in wash-off personal care product